Engineering Hierarchal Lotus Leaf-Inspired PVDF-HFP/PVDF Dual-Layer Surface Supported PVDF Composite Membrane for Desalination Via Membrane Distillation

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ABSTRACT: This study introduces a groundbreaking approach to enhance membrane distillation (MD) performance by incorporating a lotus leaf-inspired Poly (vinylidene fluoride-co-hexafluoropropene)/polyvinylidene fluoride (PVDF-HFP/PVDF) dual-layer coating onto a hollow fiber PVDF support membrane. The synergistic effect of this integrated dip-coating phase inversion approach significantly improves membrane wetting resistance characteristics, water permeability and salt rejection without compromising overall performance. Experimental results demonstrate a substantial increase in water flux from 11.55 kg/m²h–15.25 kg/m²h while maintaining exceptional salt rejection rates exceeding 99.9%. The enhanced membrane properties are attributed to the synergistic interaction between PVDF-HFP and PVDF, resulting in a hierarchical rough surface with contact angle reached up to 137.31° that effectively repels water and minimises pore wetting. This research paves the way for the development of high-performance MD membranes with improved efficiency and sustainability.

Keywords: membrane distillation, desalination, PVDF-HFP, wetting resistance, hollow fiber membrane

1. INTRODUCTION

Nowadays, population expansion, industrialisation and climate change have all contributed to an increase in freshwater demand, prompting the development of several desalination methods such as electrodialysis, reverse osmosis (RO), forward osmosis (FO) and membrane distillation (MD).¹ While these technologies have made significant strides in providing freshwater, their high energy consumption and reliance on fossil fuels pose environmental and sustainability concerns. Among these methods, MD has emerged as a promising solution to address the water-energy crisis.²

MD is a thermally driven desalination process that uses low-grade heat to efficiently produce clean water.³ Although it has a high salt rejection rate, it has challenges such membrane temperature polarisation, fouling and pore wetting.⁴ To enhance performance, fabricating the desired microporous hydrophobic membrane is crucial. To create highly hydrophobic polyvinylidene fluoride (PVDF) membranes and enhance MD performance, researchers have focused on constructing micro/nanostructures on the membrane surface and reducing its surface free energy. Inorganic particles such as titanium dioxide (TiO₂) and silicon dioxide (SiO₂) have been incorporated to build these structures, but their stability with polymer membranes poses limitations.^{5,6} Inspired by natural examples as the lotus leaf and insect repellency, super-hydrophobic surfaces have found applications in various fields, including wetting problems.⁷ The potential of super-hydrophobic surfaces to address wetting problems has triggered researchers to explore their applications in various fields. One common method to create wetting resistant surfaces is to reduce surface free energy by functionalising with low surface energy materials, such as fluorosilanes and fluoropolymers.⁵ Several studies have demonstrated improved MD performance through the engineering of repellent coating structures. The two-step procedure of non-solvent phase inversion and dip-coating was used by Xu et al. to modify polypropylene hollow fiber (HF) membranes in order to produce a superhydrophobic membrane with contact angle (CA=157°), 99.8% salt rejection and water flux up to 1.2 kg/m²h in MD.⁸ By covering the surface of a PVDF membrane with a semiinterpenetrating hydrogel coating of poly (diallyldimethylammonium chloride)/ polyacrylic acid (PDADMAC/PAA), membrane wetting behaviour in direct contact membrane distillation (DCMD) process was decreased by Ardeshiri et al.9 With a porosity of 53% and a CA of 50°, the membrane achieved a permeate flux of 5 kg/m²/h while maintaining a low conductivity of 20 µS/cm. However, polyvinylidenefluoride-hexafluoro propylene (PVDF-HFP) copolymer is more hydrophobic than PVDF due to the addition of fluoropropylene, while also maintaining tensile strength reached up to 5.7 MPa with 70.7% porosity and

30 µm thickness.¹⁰ HF membranes made from PVDF-HFP have been fabricated for DCMD, but their performance in terms of flux generally lags behind PVDF membranes.¹¹ Therefore, the synergistic combination of PVDF and PVDF-HFP is anticipated to augment anti-wetting properties, mechanical strength and DCMD performance, drawing inspiration from the lotus leaf's super-hydrophobic surface, which is achieved through advanced coating techniques.

This work employed a two-step dip-coating-phase inversion technique to develop a dual-layer lotus leaf porous PVDF-HFP/PVDF surface on the external layer of PVDF HF support membranes. This synergistic approach, combining hydrophobic fluorinated polymer PVDF-HFP and PVDF, has remained unexplored for creating excellent water-repellent surface for MD HF membranes. The impact of surface alteration on water permeability, wetting characteristics and structural features was examined using a variety of characterisation approaches. The findings of this study are anticipated to pave the way for the development of durable, water-repellent PVDF HF membranes with superior MD performance, thereby revolutionising desalination and enabling stable operation.

2. EXPERIMENTAL

2.1 Chemicals

Solvay Solexis in France provided the powdered PVDF, marketed under the name Solef®6010/1001. Sigma Aldrich (Germany) provided the N, N-Dimethylacetamide (DMAc) (99%) and PVDF-HFP pellets. Merck (Germany) supplied ethanol (>99.9%), sodium chloride (99.99% Suprapur®), polyethylene glycol 1,500 and lithium chloride. Aladdin Chemicals Co. Ltd. in China provided nano fumed silica (hydrophobic-120). During the spinning process, filtered water was used as an external coagulant.

2.2 Composite HF Membrane Fabrication

Through a dry-wet phase-inversion technique, nano-fumed silica (SiO₂)/PVDF HF support membranes were prepared according to our previous work.¹² PVDF-HFP/PVDF HF composite membranes were fabricated using a dip coating-phase inversion process. A dilute coating solution was prepared by dissolving PVDF powder and PVDF-HFP pellets in DMAc, with the specific compositions outlined in Table 1. To ensure homogeneity, a temperature of 60°C and moderate stirring for 8 h were employed. Prior to coating, the PVDF HF support membranes were cleaned with distilled water and sealed at both ends using epoxy

resin to prevent solution infiltration. The sealed HF support membranes were dipped in the dilute coating solution for 10 s, followed by immediate immersion in a coagulation bath containing distilled water. This phase inversion process, facilitated by the diffusion of water into the solution and solvent out, resulted in the formation of a porous PVDF/PVDF-HFP coating layer. The coagulation bath was refreshed with distilled water every 8 h for a total of 3 cycles. Finally, the coated HF composite membranes were dried at 25°C. To investigate the influence of PVDF-HFP concentration on membrane properties, a series of composite membranes were prepared with varying concentrations of PVDF-HFP in the coating solution. These membranes were designated as HF-P0, HF-P1.5, HF-P3 and HF-P4.5, respectively. The uncoated PVDF HF support membrane served as a control and was labeled as HF-0.

Compositions (wt.%)	Membrane samples				
	HF-P0	HF-P1.5	HF-P3	HF-P4.5	
PVDF	2	2	2	2	
PEG-1500	33	3	33	33	
DMAc	65	63.5	62	60.5	
PVDF-HFP	0	1.5	3	4.5	

Table 1: The PVDF-HFP diluted coating formulations

Note: PEG-1500 = polyethylene glycol 1500

2.3 Characterisation Methods

The coating solution viscosity was conducted with the support of rotary Brookfield Viscometer (RVDV-11+P, USA) at ambient temperature. First, the dope to be measured was poured into the examining container and a proper spindle attached and dipped into the solution. Then, an appropriate speed was chosen (50 rpm), and spindle size (27) kept rotating for 5 min until a steady value attained. The viscosity was estimated based on the rotation factor, spindle factor and rotation speed. Five readings per sample were recorded and the average was recorded. Surface morphology was analysed using scanning electron microscopy (SEM). ImageJ software was used to measure the apparent thickness of the coating layer of the HF membrane. Contact angle measurements, porosity determination, pore size analysis and iquid entry pressure (LEP) measurements were conducted to characterise hydrophobicity, porosity, pore size and liquid entry pressure according to procedures explained in detail in our previous work.^{12,13} Experimental errors were minimised by collecting and averaging multiple data points for all samples.

2.4 MD Performance Test

Four HF membranes, each 20 cm long, were used to prepare an MD module. 18 cm and 0.0015 m² were the effective length and area, respectively. DCMD experiments were conducted with a 3.5 wt.% sodium chloride (NaCl) feed stream solution (hot side) and deionised (DI) water (cold side) as permeate stream as illustrated in Figure 1. Both streams were maintained at 250 mL/min and controlled temperatures of 70°C and 18°C. A computerised weighing balance was used to measure permeate production, and further information are provided in our previous study.¹² Permeate flux (J, kg/m²h) and NaCl rejection (SR, %) were calculated using the provided equations.

$$J = \frac{\Delta W}{A.\Delta t} \tag{1}$$

Where Δt is the time interval, A is the effective membrane area of HF (m²) and ΔW is the increase in weight of permeate obtained (kg).

$$SR(\%) = \left[1 - \frac{C_p}{C_f}\right] \times 100 \tag{2}$$

The aforementioned equation, in which C_p is the permeate concentration and C_f is the feed concentration (NaCl) solution, g/L, was used to compute the salt rejection, *SR* (%) using a conductivity meter.



Figure 1:Schematic set up of DCMD unit.

3. **RESULTS AND DISCUSSION**

3.1 Morphology

The surface morphology of the HF membranes was examined using SEM as shown in Figure 2. SEM images revealed that a thin coating layer, exceeding 10 μ m in thickness, was deposited on the outer surface of the PVDF HF support membrane after applying the diluted PVDF coating solution. The incorporation of PVDF-HFP polymer into the diluted PVDF coating solution led to a rougher surface on the HF membrane. This roughness was observed to be greater than that of both the uncoated PVDF support membrane Figure 2(a) and the membrane coated with only the PVDF dilute solution Figure 2(b). However, all HF membranes, however, had an obvious porous coating layer on the exterior, as depicted in Figure 2(b–e). The formation of a semi-continuous porous structure on the exterior surface of the membrane was attributed to the properties of the PVDF-HFP dilute solution and the dip-coating phase separation technique.



Figure 2: Surface morphology images (SEM) (a) HF-0, (b) HF-P0, (C) HF-P1.5, (d) HF-P3 and (e) HF-P4.5. The scale bars in the micrographs correspond to 100 μm.

The moderate viscosity and short coating time hindered the solution's penetration into the PVDF membrane pores. This resulted in the preferential formation of the semi-continuous structure during the subsequent phase inversion process. These unique structures significantly increased the surface roughness of the PVDF membrane. Similar to the bulges on a lotus leaf, these structures facilitated the maintenance of spherical water droplets on the outer surface of the membrane.¹⁴ This, in turn, enhanced the hydrophobicity of the membrane. Nevertheless, an increase in the concentration of PVDF-HFP in the coating solution led to a corresponding thickness increase for the resulting coating layer. This is evident from the results presented in the Table 2. Thicker coatings can potentially hinder mass transfer and water flux during MD.¹⁵ Despite the variation in coating thicknesses, which within the range from 2 μ m–739 μ m, all coatings were deemed suitable for MD applications.¹⁶

3.2 Membrane Wettability Resistance

To enhance wetting resistance and flux for MD, membranes with hydrophobic properties similar to the self-cleaning lotus leaf are crucial. The wetting of hydrophobic or super-hydrophobic porous membranes adversely affects mass transfer, leading to a decline in permeate flux.¹⁷ However, due to the high electronegativity of fluorine, fluoropolymers such as PVDF-HFP exhibit low surface energy, thereby limiting their affinity for other materials.¹⁸ Hydrophobicity was assessed through the measurement of both static and dynamic water contact angles. The temporal variation of water contact angle (WCA) for diverse membrane types, as depicted in Figure 3 and Figure 4, provides a comparative analysis over a 240 s interval. The pristine uncoated PVDF membrane (HF-0) exhibited a WCA of $105.37^{\circ} \pm 0.34$. However, after applying the PVDF composite coating (HF-P0), the WCA increased to $124.75^{\circ} \pm 1.6$. This enhancement in hydrophobicity is attributed to the coating itself. Incorporating the PVDF-HFP polymer into the diluted PVDF coating solution further improved the anti-wetting and lotus leaf properties of the membrane. A progressive increase in PVDF-HFP concentration within the dilute coating solution resulted in a corresponding gradual elevation of the water contact angle (WCA), reaching a maximum value of $137.31^{\circ} \pm 1.5^{\circ}$ for HF-P4.5. These results demonstrate the significant impact of the coating and the incorporation of PVDF-HFP on the membrane's hydrophobicity.



Figure 3: Static WCA values for different HF membranes as function of PVDF-HFP concentration.



Figure 4: Dynamic WCA values for different HF membranes as function of PVDF-HFP concentration.

Partial or complete pore wetting in MD can significantly reduce permeate flux and compromise membrane performance.¹⁹ LEP is a critical parameter that directly influences the wettability of various HF membranes. Membrane wetting ensues when the transmembrane hydrostatic pressure surpasses the LEP threshold.

Therefore, by measuring LEP, we can assess the effectiveness of the coating in maintaining the membrane's hydrophobic properties and preventing wettingrelated issues. Table 2 presents the LEP result for both the uncoated PVDF HF support membrane and the modified PVDF-HFP/PVDF HF membranes. A distinct upward tendency was observed in the LEP values of all coated membranes, ranging from 170 kPa for the unmodified membrane (HF-0) to a peak of 185 kPa for the PVDF-HFP/PVDF (HF-P3) composite membrane. Subsequently, a slight reduction in LEP was noted for the highest PVDF-HFP loading (HF-P4.5), which may be attributed to increased viscosity within the coating matrix layer.²⁰ The synergistic interplay between PVDF and PVDF-HFP within the coating layer is believed to contribute significantly to the enhanced lotus leaf-inspired wetting resistance and overall membrane performance.

3.3 Membrane Structural Parameters

Water vapour flux in MD is greatly influenced by the membrane's structural parameters, including pore size, porosity and thickness. Small pore sizes, high porosity and reduced thickness are essential for enhancing wetting resistance and permeability.²¹ Table 2 presents the structural characteristics of diverse modified PVDF HF membranes. While minor variations in mean pore size were observed among the coated HF composite membranes and uncoated PVDF HF support membrane, all values remained within a narrow range of 0.162 μ m–0.223 μ m. These findings demonstrate that the surface alteration employing the diluted PVDF-HFP/PVDF coating solution layer did not induce substantial alterations to the pore diameter or structural property of the membrane.

To assess the influence of the diluted coating solution on the external surface properties of HF membranes, porosity and coating thickness measurements were further conducted. As outlined in Table 2, the porosity values ranged from 81.58%-86.1%, indicating suitability for MD applications. While high porosity is desirable for maximising permeate flux, it can also compromise mechanical strength, making membranes more susceptible to leakage or damage.²² Table 2 reveals an inverse relationship between PVDF-HFP loading and membrane porosity, with a slight decrease in porosity observed at 4.5 wt.% PVDF-HFP. However, these results indicate that the application of a thin coating layer to the surface of a PVDF support HF membrane exerts a minimal influence on the pore structure of the resulting membranes. The coating layer thickness, initially measured at 13.45 µm without PVDF-HFP incorporation, exhibited a gradual increase with the addition of PVDF-HFP, reaching a thickness of 35.89 µm at a 4.5 wt.% PVDF-HFP concentration. While an increase in coating thickness may potentially mitigate thermal conductivity and reduce heat loss during the MD

process, it may also result in diminished permeability due to the hindered mass transfer and diffusion of water vapour molecules through the thicker coating layer. However, the narrow pore sizes of the modified PVDF composite HF membranes effectively mitigate wetting and additional mass transfer resistance, ensuring sustained water vapour flux and salt rejection throughout the MD process.²³

Membrane	LEP (KPa)	Porosity (%)	Average pore size (µm)	Coating thickness (µm)	Viscosity of the coating solution (cP)
HF-0	170 ± 0.91	86.10 ± 1.10	0.162 ± 0.13	-	-
HF-P0	173 ± 0.75	85.22 ± 0.93	0.175 ± 0.08	13.45 ± 0.15	54.20 ± 0.31
HF-P1.5	178 ± 0.57	84.76 ± 0.55	0.188 ± 0.05	18.35 ± 0.23	56.10 ± 0.02
HF-P3	185 ± 0.75	84.22 ± 0.93	0.195 ± 0.08	22.45 ± 0.15	58.20 ± 0.25
HF-P4.5	179 ± 0.34	81.58 ± 1.89	0.223 ± 0.02	35.89 ± 0.25	61.35 ± 0.09

Table 2: Characteristics of different modified HF membranes

3.4 DCMD Separation Performance

The synergistic impact of PVDF-HFP/PVDF coating film applied to the outer surface of the PVDF support HF membrane resulted in a significant enhancement of both NaCl rejection and permeate flux during the DCMD process, as depicted in Figure 5. This enhancement can be ascribed to the existence of fluorine, which altered the membrane's surface properties and induced water-repellent behaviour. All coated PVDF-HFP/PVDF membranes exhibited NaCl rejection rates consistently exceeding 99.9%, except for HF-P4.5, which had a rejection rate of 98%. The layer of PVDF coating (HF-P0) applied to the support PVDF HF (HF-0) membrane resulted in an increase in water permeation flux from 11.61 kg m⁻² h⁻¹ to 12.15 kg m⁻² h⁻¹. The subsequent dosing of PVDF-HFP within the layer of PVDF coating further improved water permeation flux, reaching a peak value of 15.25 kg m⁻² h⁻¹ for HF-P3 membrane. However, a 4.5 wt.% PVDF-HFP dose in the PVDF layer for HF-P4.5 resulted in a decrease in permeation water flux to 11 kg m⁻² h⁻¹. The high PVDF-HFP loading in HF-P4.5 resulted in a significantly higher viscosity coating solution (61.35 cP), leading to a thicker membrane with reduced porosity, as confirmed by the data in Table 2. This combination of increased thickness, decreased porosity, and slightly lower LEP collectively contributed to the observed reduction in permeate flux and salt rejection.¹⁴ These results underscore the potential of the synergistic effect of the dual porous and hydrophobic PVDF-HFP/PVDF coating layer, applied via the dip coating-phase inversion process, to significantly enhance water permeability in DCMD while maintaining robust desalination performance.



Figure 5: Desalination performance of modified PVDF-HFP membranes in DCMD.

4. CONCLUSION

The incorporation of PVDF-HFP into the PVDF coating layer via the integrated dip-coating phase separation approach significantly augments MD performance. This enhancement is attributed to morphological changes, as evidenced by SEM, LEP, pore size, contact angle and porosity analysis. Remarkably, a mere 3 wt.% PVDF-HFP yields an optimal MD membrane with a water flux of 15.25 kg/m²h and high salt rejection (>99.9%). These findings contribute to advancing MD technology by demonstrating the effectiveness of PVDF-HFP/PVDF dual layer through constructing a rough and highly hierarchical water- repellent surface of HF membranes.

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