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Enhanced Water Purification Using Hydrophobic PVDF-HFP Membranes in DCMD: Synthesis and Performance Analysis

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This research paper presents a comprehensive investigation into the purification of artificial marine solution (brine) using hydrophobic polyvinylidene fluoride-co-hexafluoropropylene (PVDF-HFP) flat sheet membranes. The membranes were prepared through a Phase inversion process involving the dissolution of the polymer in dimethylacetamide (DMAc) as the solvent. The synthesized membranes were subjected to a thorough characterization, including analyses of morphology, porosity, and hydrophobicity. In the experimental setup, DCMD was employed as the desalination technique, utilizing the hydrophobic nature of the PVDF-HFP membranes to facilitate efficient water vapor transport while preventing undesired wetting. The efficacy of the prepared membrane was evaluated in treating saline solution containing 30g/L of NaCl. The initial concentration of total dissolved solids (TDS) of prepared brine was elevated, measuring at 27784 mg/L. The study found that the PVDF-HFP membranes attain salt rejection of about 99% for the feed solution at a water flux of over 11.11 L/m2h which accounts for 34% for recovery. The membrane exhibited consistent permeability throughout the 24-hour continuous desalination process, showcasing its stability. The findings not only advance our understanding of membrane-based desalination processes but also provide a foundation for the design and optimization of membrane systems for broader applications in water treatment and resource management.

Keywords: Permeability, Membrane distillation, Phase inversion, Hydrophobic polymer.

1. Introduction

Freshwater scarcity is a direct result of an increasing worldwide population and higher agricultural and industrial water consumption; approximately 1.3 billion people lacked a source of pure water (Mishra,2023),and an additional 2.3 billion reside in regions with shortages of water (Sharma and Nayak,2024).Developing nations with swiftly growing populations and a shortage of energy appears to be particularly impacted. In light of the fact that rising water needs cannot be met solely with natural clean water, the global community has resorted to desalinization, the heavy on energy procedure, as a water-based complement. Arid areas, such as the Arabian Peninsula, which have extremely limited access to naturally sources of freshwater, obtain the majority of their water requirements from desalination (Al-Addous et al.,2024).Over the past few years, membrane processes have progressively supplanted thermal-based desalination technologies, which are more energy intensive. Presently, RO (reverse osmosis) represents nearly 60% of desalination deployments worldwide (Anis et al.,2019; Drioli et al.,2015). Despite having approximately half of the world’s distillation capacity, the region of the Middle East continues to rely on thermal desalination for its water supply because fossil fuels are readily available and the local feed water is of low quality (Greenlee et al.,2009). Therefore, the development of more environmentally friendly technologies for desalination is of the utmost importance. Potentially more unsustainable distillation methods have been considered suggested throughout the years and are the subject of intensive research at present. One such method is the use of membrane distillation (MD), which is a hybrid thermal-membrane procedure that separates water from saline via a microporous membrane using an atmospheric pressure gradient caused by temperature. MD is an appealing distillation method for a number of causes, which includes its potential to reduce energy costs, resistance to fouling of membranes, and near-zero saline production due to its resistance to osmotic pressure change (Alkhudhiri et al.,2012; Boubakri et al.,2024).The development of contemporary applications relying on artificial membranes requires polymers with distinctive properties. Polymeric materials must exhibit robust resistance to acids, bases, oxidants, and reductants, along with the ability to withstand high pressures and temperatures. Additionally, these materials should possess specific chemical properties to optimize permeation flux and achieve an appropriate solute separation factor for the envisioned applications (Park et al.,1999; Ali M.S.M,2005). Over the last four decades, membrane-based separation technologies have experienced significant growth, evolving into a thriving industry. In 2019, this sector contributed to a substantial business valuation of USD 13.5 billion, with projections anticipating a further increase to USD 19.6 billion by the year 2025 (Tang et al.,2021). The Membrane Distillation (MD) process inherently exhibits a lower susceptibility to fouling when juxtaposed with pressure-driven membrane operations (Talukder et al. 2024) .This is attributed to several factors: (a) the pores in MD are comparatively larger than those in Reverse Osmosis (RO) and Ultrafiltration (UF), (b) the process liquid cannot cause wetting of the membrane, limiting fouling layers to deposition solely on the membrane surface rather than within the membrane pores, and (c) owing to the process's low operating pressure, any aggregates deposited on the membrane surface are less compact, causing only minimal impact on transport resistance. Theoretically, MD is envisioned as 100% impenetrable to non-volatile components, including macromolecules, colloidal species, and ions (Saidi et al.,2021). PVDF-HFP, a copolymer, has garnered attention as a potential material for membrane applications in contrast to poly (vinylidene fluoride)

(PVDF), PVDF-HFP exhibits reduced crystallinity due to the inclusion of hexafluoro propene comonomer (HFP) in the main backbone, resulting in a notable increase in the amorphous phase content. Additionally, the presence of HFP groups is believed to augment the fluorine content, contributing to PVDF-HFP possessing more effective hydrophobic chains (Stephan and Teeters,2003; Stephan et al.,2004; Cao et al.,2006; Sundaram and Subramania,2007; Abid et al.,2024). The majority of investigations into the preparation of PVDF-HFP membranes involve the use of hazardous dipolar aprotic solvents, such as N-methyl-2-pyrrolidone (NMP), N, N-dimethylformamide (DMF), and N, N-dimethylacetamide (DMA). This choice is attributed to the significant solvent capability for dissolving polymers, high boiling points, and effective miscibility in water.

Song et. al reported the polymer-solvent compatibility order as follows: TEP > NMP > DMAc > DMF > DMSO where Triethyl phosphate (TEP), N-methylpyrrolidone (NMP), dimethylacetamide (DMAc), dimethylformamide (DMF), dimethylsulfoxide (DMSO) have been studied (Song et al.,2018).

Wongchitphimon et al. demonstrated that incorporating PEG into the PVDF-HFP/N-methyl-2-pyrrolidone (NMP) solution led to a system that was thermodynamically less stable in its interaction with water, facilitating swift phase separation during the phase inversion process. The introduction of 3 wt.% PEG into the polymer solution resulted in the enlargement of finger-like macro-voids in the membrane. This enlargement was observed to escalate with the increase in PEG molecular weight, ranging from 200 to 600 and 6000 kDa, consequently improving the water permeability of the membrane (Wongchitphimon et al.,2011). Another report investigated production of Flat sheet membranes of Poly (vinylidene fluoride-hexafluoropropylene) (PVDF-HFP) for applications in aqueous membrane distillation (MD) by employing triethyl phosphate (TEP) as a less-toxic solvent through phase inversion. The study explored PVDF-HFP concentrations ranging from 10 to 15 wt.%, revealing that varying polymer concentrations resulted in membranes with distinct surface structures and performance characteristics. Additionally, the utilization of different coagulation bath compositions played a crucial role in membrane fabrication, influencing the performance of the membranes in the context of membrane distillation (Fadhil et al.,2016).

In this study, porous membranes of PVDF-HFP were created using DMAc via phase inversion technique and subjected to testing in direct contact membrane distillation (DCMD).The performance of fabricated PVDF-HFP membrane was investigated with a focus on morphology, pore size, porosity, and thickness. The prepared membranes were evaluated for treatment of brine.

2. Experimental

2.1 Procurement of Chemicals

To prepare membranes, PVDF-HFP copolymer (Mw = 455,000, Sigma-Aldrich) and N, N-dimethylacetamide (DMAc, >99.8%, Merck)) as the solvent are used. Sodium chloride (NaCl,>99%) was purchased from CDH (Central Drug House) used for the preparation of brine. Distilled water was used throughout this study.

2.2. Membrane preparation

Fabrication of membrane have been done via phase inversion technique. Dope solutions with varying ratio of PVDF-HFP and DMAc were prepared by dissolving the copolymer P(VDF-HFP) in the solvent (N, N-Dimethylacetamide (DMAc) using magnetic stirrer at 60˚C for 6 h. The homogeneous solutions were achieved in 8 hours. After the degasing time of two hours, the molding mix was evenly distributed across a glass plate, using a molding knife with dimensions of 2.00×10−4 meters. Once it had been exposed to air for 20 seconds, the glass plate was placed in a container filled with distilled water. The filter membranes were then submerged in this distilled water. Subsequently, the membranes were left to air dry to eliminate any excess moisture. The nascent membranes were immersed in a non-solvent water bath at 15˚C. The Polymer to Solvent proportion in prepared membrane are 7.96% and 92.04% respectively

2.3. Membrane Distillation Experimentation

The DCMD experiments were carried out across a range of temperatures (ranging from 40°C to 80°C) and feedwater flow rates (ranging from 80 mL/min to 100 mL/min). The process of distillation time and heat input was systematically investigated, with each experiment being replicated 10-12 times, each cycle spanning 4 hours, to ensure the reliability of the results and to calculate the water flux. The performance of the membranes was evaluated based on their permeation capabilities and their ability to reject NaCl, as indicated by Eqs. (1) and (2).

$$\text{Permeation}\left(\frac{\text{kg}}{\text{m}^2\text{h}}\right) = \frac{\text{Wtp}}{\text{Axt}} \qquad (1)$$

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

where Wtp is the permeate weight, A is the membrane area, t is the experiment time, Cp is the permeate concentration and Cf is the feed concentration.A schematic diagram illustrating the design of the Distillation Membrane Distillation (DCMD) system is presented in Figure 1.

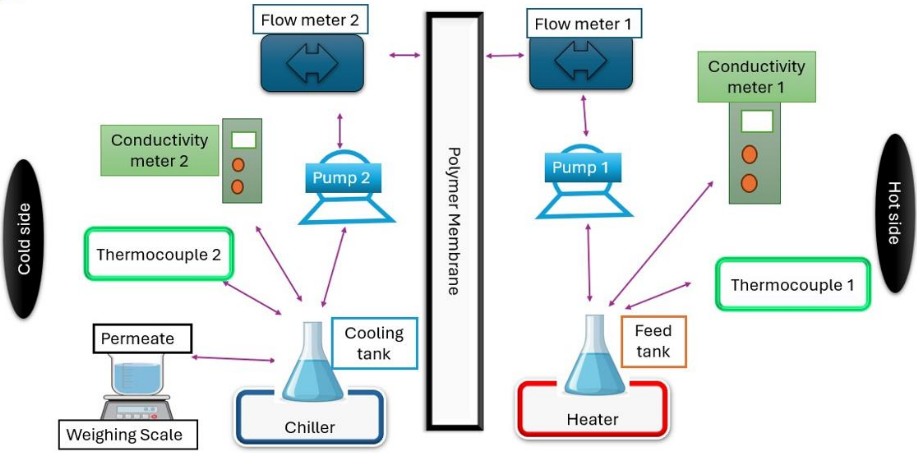

Figure 1: Schematic DCMD set up.

2.4. Membrane characterization

2.4.1. Membrane porosity

The membrane porosity was calculated according to the following Eq. (3):

$$\text{Porosity (\%)} = \frac{\dfrac{wt_w - wt_d}{\rho_k}}{\dfrac{wt_w - wt_d}{\varphi_k} + \dfrac{wt_d}{\rho_p}} \qquad (3)$$

where wt_w is the membrane wet weight, wt_d is the membrane dry weight, ρ_k is the kerosene density (0,82 g/cm^3) and ρ_p is the polymer density (0,98 g/cm^3). For each porosity measurement, five different parts of the same membrane were immersed with kerosene for about 48 h. The mass before and after immersing in kerosene was obtained using a precision balance.

2.4.2. Membrane thickness

Membranes thickness was measured using micrometer with a precision of ±0.001 mm. At least three regions of membrane were measured and the average value of membrane thickness was taken into account.

2.5 Membrane Characterization

2.5.1 Scanning Electronic Microscopic (SEM)

The morphology of the composite membranes was studied by High Resolution Field Emission Scanning Electron Microscope with EDS (FE-SEM) (7610F Plus/JEOL, Resolution of 0.8nm @ 15kV 1.0nm @ 1 kV). The density of the samples (expressed as mass (mg) per volume (cm^3)) was measured by weighing an area of the produced membranes and measuring the average thickness based on the SEM cross-section micrographs and using Image J software.

2.5.1 Differential scanning calorimetry (DSC)

Process was carried out under a nitrogen atmosphere with Discovery 25/ TA Instruments Waters, using samples in aluminum pans. The measurements were conducted from 25 to 400˚C at a heating rate of 5˚C/min.

2.5.2 X-ray diffraction (XRD)

It is a versatile analytical technique used to analyze physical properties such as phase composition, crystal structure, and sample orientation. For this purpose, the Multipurpose Versatile XRD System (Smart Lab 3kW, Rigaku) was employed.

3. Results and discussion

3.1 Feedwater Characterization

Table 1 and Figure 2 presents the water quality parameters of the prepared brine as collected and after treatment with PVDF-HFP membrane through DCMD experimental setup. It is evident that the total dissolved solids (TDS) are extremely high. The predominant inorganic ions include chlorine, calcium, magnesium and fluorine. The table compares the quality of brine before and after treatment with a pristine against the IS 10500:2012 standards. The pH levels ranged from 8.16 in untreated brine to 7.75 in the polymer treated water, within the acceptable limit of 6.5-8.5.Total hardness and calcium levels significantly decreased from 1020.86 to 155.12 mg/L, respectively, after treatment with fabricated membrane meeting the acceptable limits. Magnesium and chloride levels also dropped markedly from 107 mg/L to 20.67 mg/L, respectively. Total alkalinity, total dissolved solids (TDS) remarkably to acceptable limits after treatment. Fluoride levels remained consistent, within the acceptable range, and nitrogen nitrate levels also decreased significantly. Overall, the treatments, effectively reduced contaminants to meet or exceed the acceptable limits set by IS 10500:2012 as presented in figure 7. The fabricated membrane performance was comparable to that of membranes reported in literature, as presented in Table 3 and Fig.6.

Table 1: Chemical characterization of the water samples before and after treatment with polymer membrane.

S.NO	Test Parameter	Feed Water (Brine)	Permeate Water (Brine)	Specification as per IS 10500:2012 Requirement (Acceptable Limit)	Permeable Limit in the absence of alternate source
1	pH	8.16	7.75	6.5-8.5	No relaxation
2	Total Hardness as $CaCO_3$ (mg/l)	1020.82	155.12	200	600
3	Calcium as Ca (mg/l)	232.46	28.06	75	200
4	Magnesium as Mg (mg/l)	107	20.67	30	100
5	Chloride as Cl (mg/l)	2340	384.35	250	1000
6	Total Alkalinity as $CaCO_3$ (mg/l)	155	40	200	600
7	Total Dissolved Solid (mg/l)	27784	3988	500	2000
8	Sulphate as SO_4(mg/l)	47.24	76.08	200	400
9	Fluoride as F	0.85	0.80	1.0	1.5
10	Nitrogen Nitrate as NO_3	0.53	0.22	45	No relaxation

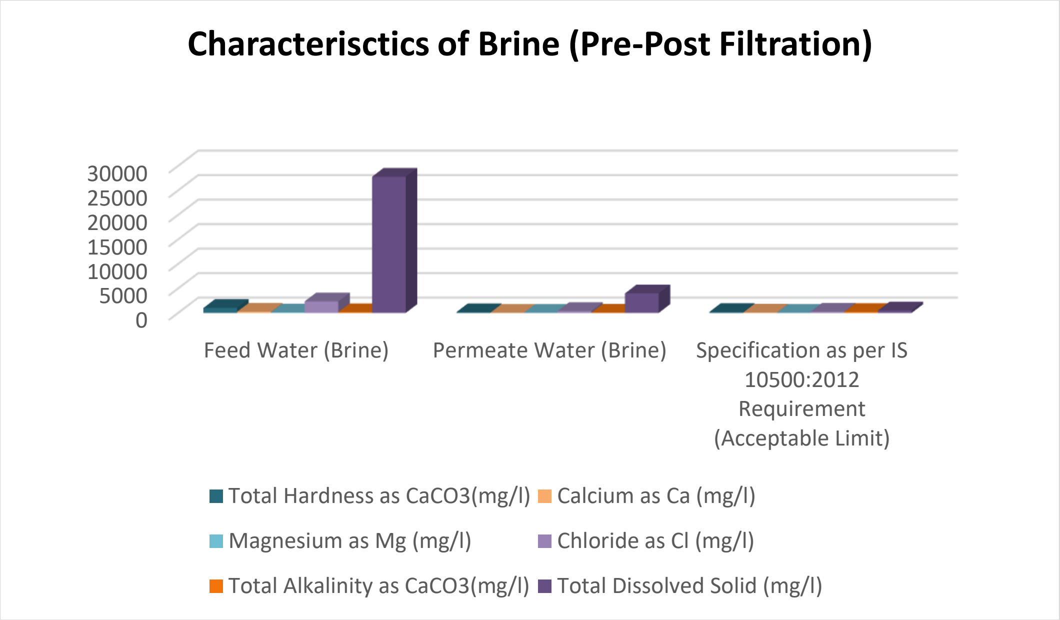

Fig. 2: Comparison of feed water and permeate (brine)

3.2 TDS, Salinity and conductivity as a function of operational time

This section examines the behaviors of permeate flux, feed TDS, conductivity and salt rejection percentages over time when using PVDF-HFP membranes, presented in Figure 4(a),4(b). Cath et al. (2004) investigated the performance of direct contact membrane distillation (DCMD) with sodium chloride and synthetic feed salt solutions at 40°C, using membranes of varying thicknesses. They found a 99.9% salt rejection rate and observed that feed salt concentration had only a minor impact on water flux. Similarly, Fard and Manawi (2014) explored seawater desalination using hydrophobic PTFE membranes in a DCMD process over 17 hours, reporting a 99.9% salt rejection rate for all tested salts. Despite this high rejection rate, low fluxes were recorded at all temperatures with the PTFE membrane. In contrast, commercial polyethylene (PE) membranes, known for their intrinsic hydrophobicity, low surface energy, and sponge-like morphology, could enhance flux. Their porosity, appropriate pore size, and reasonable wetting resistance make them suitable for the MD process. At higher feed flow rates, PE membranes can improve permeate flux and reduce temperature polarization effects (Zuo et al., 2016). In this study, after 24 hours (4 hour per cycle) of operation, the permeate TDS concentration remained well below the WHO's permissible limit of 1000 mg/L for drinking water quality (WHO, 2011). Figure 4 illustrates the behavior of permeate flux and conductivity as a function of operating time using a PVDF-HFP membrane. The results show that the conductivity and salinity changes, with average values ranging between 2.82 and 3.02 $L/m^2 \cdot h$. This suggests that DCMD is more advantageous for desalinating salt solutions compared to pressure-driven membrane techniques, which typically exhibit a significant decline in permeate flux after a short operating period (Olatunji et al.,2024)

Regarding water quality, the permeate conductivity showed a slight decrease from 228 µS/cm to 205 µS/cm, with stable permeate quality over time. In some cases, the evaporation of volatile components from the feed solution can alter the composition of the solution, potentially reducing the overall concentration of dissolved ions and lowering conductivity. Also as the process continues, a concentration gradient can form near the membrane surface, leading to the accumulation of salts and other solutes in the feed solution. This buildup can reduce the effective concentration of solutes that reach the permeate side, thereby lowering the conductivity of the permeate. Table 2 and figure…The minimal increase in permeate flux and the decrease in conductivity indicate that the DCMD process maintains high stability during operation, with no significant issues such as membrane wettability or fouling, which aligns with previous studies (Tai et al.,2023). Consequently, a running time of 4 hours was deemed sufficient for the remaining experiments.

Table 2: Variation of permeate conductivity and salinity with time.

S.No	Operation time (h)	Conductivity(µs/cm)	Salinity(mg/l)
1	1	228	112
2	2	210	104
3	3	206	98
4	4	205	86

3.2 Scanning Electron Microscopy (SEM) Analysis

This section presents the results of the Scanning Electron Microscopy (SEM) analysis, featuring high-resolution images and elemental mapping that reveal the structural characteristics of the sample. The morphology of prepared flat sheet membrane pre and post filtration were studied and reported in Fig. 3.

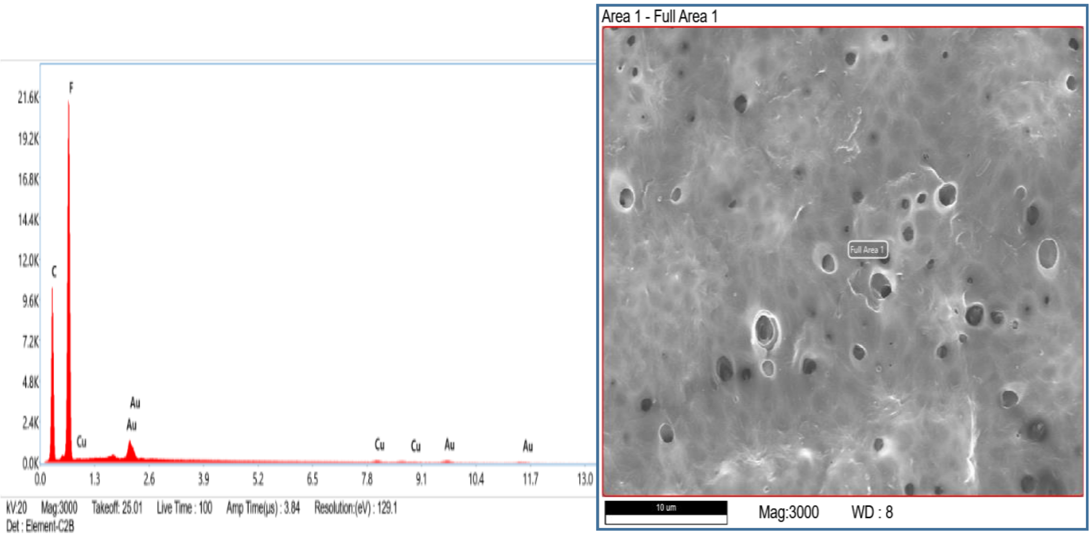

Fig. 3: Morphology of prepared flat sheet

This occurs because particles and contaminants from the filtered solution can become trapped within the pores of the membrane, leading to clogging and a reduction in the effective pore size. Over time, this can result in decreased porosity and reduced filtration efficiency.

3.3 Differential scanning calorimetry (DSC) Studies

The pure PVDF-HFP curve, illustrated in Figure 4(a), shows two distinct peaks at 119°C and 135°C, corresponding to the α-phase and β-phase, respectively. This double melting peak is attributed to the polymorphic structure of the material and is also linked to the recrystallization of the molten polymer and the presence of imperfect crystals in pure PVDF-HFP. Additionally, it may be associated with variations in bonding arrangements, such as "head-to-head" or "tail-to-tail" configurations within the PVDF-HFP membranes, which influence their thermodynamic behavior and crystalline phase formation (Dillon et al., 2006; Malmonge et al.,2003). Further studies suggest that this curve represents not a single peak, but rather a drop due to phase transition (Merlini et al.,2014). PVDF-HFP (polyvinylidene fluoride-co-hexafluoropropylene) membranes experience a decrease in enthalpy after the filtration process as shown in 4(b), it typically indicates a change in the membrane's internal energy or phase structure. This decrease could be attributed to several factors, such as the absorption or interaction with filtered substances, the loss of solvent or plasticizer during filtration, or a reduction in crystallinity due to physical stress or fouling. As enthalpy is related to the thermal and physical properties of the membrane, a decrease might also suggest that the membrane's structure has become less ordered or that it has undergone some degree of compaction or fouling, affecting its thermal behavior. Such changes could impact the membrane's performance, including its permeability, mechanical strength, and overall filtration efficiency.

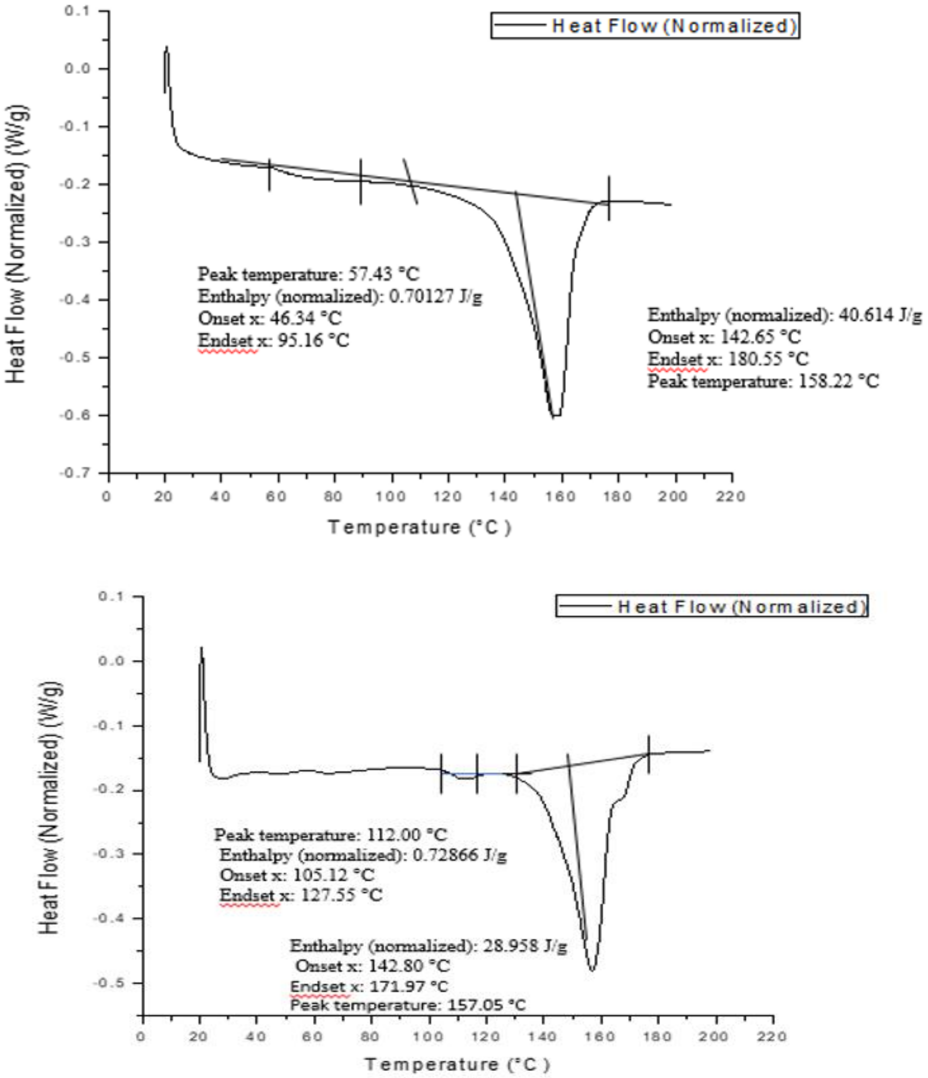

Fig.4(a),4(b): DSC thermographs for PVDF-HFP membranes (pre-post filtration) for brine as feed.

3.4 X-ray Diffraction (XRD) Studies

XRD diffractograms of PVDF-HFP membrane recorded in 2θ range from 5 to 85˚ C are presented in Fig.5(a),5(b).Generally, when a polymer has a significant crystalline region, the X-ray diffraction peak is sharp and its intensity is high, while an amorphous polymer exhibits a broader peak (20). Based on the X-ray diffraction measurements displayed in Fig. 8, pure PVDF–HFP exhibits diffraction peaks at 2θ = 19.79° and 26.61° due to the presence of form crystals (Abbrent et al.,2001). The presence of diffraction peaks at 2θ=18.3˚ and 20.1˚ corresponding to α (100) and α (020) characteristic for the α-form phase crystal of PVDF-HFP was found (Kimura N, Sakumoto T,2024)

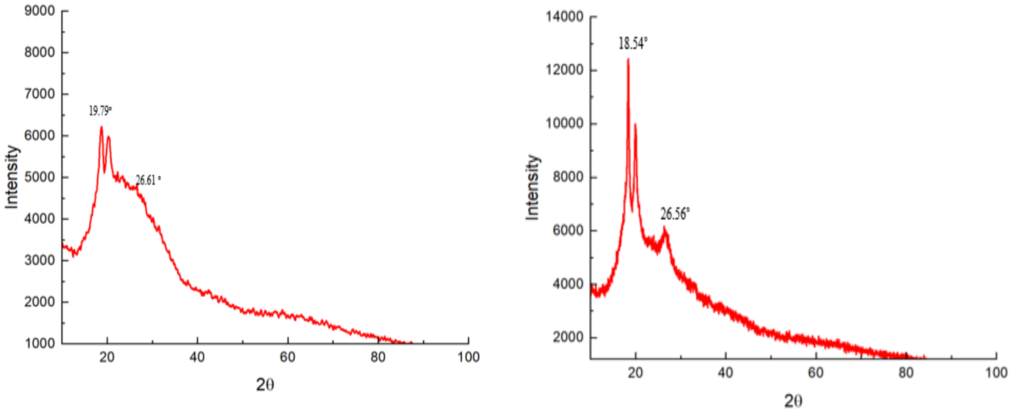

Fig. 5(a),5(b): XRD graphs of PVDF-HFP membranes for brine as feed.

4. Conclusion

This research investigates the enhancement of water purification using hydrophobic PVDF-HFP membranes in Direct Contact Membrane Distillation (DCMD). The study focused on synthesizing these membranes and evaluating their performance with respect to Total Dissolved Solids (TDS), flux, and salt rejection for both brine and faucet water.

The synthesized PVDF-HFP membranes exhibited superior hydrophobic properties, which significantly improved their performance in DCMD applications. The results demonstrated that these membranes achieved high salt rejection rates, consistently exceeding 99.5% for both brine and faucet water. This exceptional salt rejection underscores the effectiveness of PVDF-HFP membranes in removing dissolved salts and other impurities from the water.

In terms of flux, the PVDF-HFP membranes showed promising results, with higher permeate fluxes compared to traditional membranes. This improvement in flux was particularly notable under various operational conditions, highlighting the membranes' capability to enhance water production efficiency.

Furthermore, the performance analysis indicated that the membranes maintained stable TDS levels in the permeate, well below the permissible limits set by drinking water quality standards. This consistent performance demonstrates the membranes' reliability in producing high-quality purified water.

5. Future Directions and Research Opportunities:

While this study provides a foundation for understanding the potential of PVDF-HFP membranes in DCMD, there exist avenues for further exploration. Future research may delve into optimizing membrane fabrication parameters, assessing the membrane's response to varied feedwater compositions, and scaling up the system for practical applications. Moreover, a holistic approach should involve interdisciplinary collaboration to address environmental considerations, socio-economic impacts, and community engagement aspects in deploying such technologies.

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Statements & Declarations

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Author Contributions: All authors contributed to the study conception and design. Data collection was performed by Ms. Rashmi Kakkar. All authors contributed in the data analysis and manuscript preparation. All authors read and approved the final manuscript.

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