Optimization of Zeolite Incorporation on PVDF-Based Mixed Matrix Membrane for Water Desalination

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**Abstract.** Seawater desalination has become a crucial solution to address the global water crisis, especially in regions with limited freshwater resources. Membrane technology has rapidly advanced as an efficient method in the desalination process. Among previous types of membranes, polyvinylidene fluoride (PVDF) stands out due to its superior mechanical and chemical properties. This study focuses on the development of PVDF membranes modified with the addition of Zeolite Y to enhance desalination performance. Zeolite Y, with its well-defined pore structure and high adsorption capacity, is expected to improve the hydrophilicity and selectivity of PVDF membrane. This research explores the optimization of porous filler-polymer composition of MMMs to achieve high performance of seawater desalination process. PVDF membrane was prepared though phase inversion with zeolite Y loadings from 0.5, 1, 1.5 wt%. PVDF/zeolite Y MMMs exhibits several significant advantages, including increased water flux, ion selectivity, and fouling resistance. Experimental studies indicate that zeolite Y’s loading to PVDF membrane can enhance water flux and salt rejection compared to PVDF Neat membrane. These results suggest that 1% loading of zeolite Y incorporated in PVDF represents the most compromise solution for water desalination. These findings not only contribute to the development of desalination technology but also offer practical solution to achieve future clean water provision.

**Keywords:** Mixed Matrix Membrane, PVDF Membrane, Seawater Desalination, Zeolite-Y.

# INTRODUCTION

Access to clean water is one of the most urgent challenges that needs immediate resolution. Clean and healthy water supply is essential for basic human needs such as drinking, cooking, washing, and sanitation. While access to water and sanitation is often taken for granted in developed countries, it remains a significant issue for many in Indonesia. Between 2010 and 2018, the demand for water for urban and agricultural purposes saw the highest increase, accounting for 72% of the total water demand in Indonesia. The need for clean water is still met through groundwater sources. Unfortunately, groundwater represents only 0.5% of the Earth's total water supply. Overall, 97% of the Earth's water is salt water, 2.5% is locked in polar ice caps, glaciers, and the atmosphere, leaving only about 0.5% accessible for human use in rivers and groundwater[1–3]. With industrial development, freshwater from rivers and lakes has become heavily polluted due to industrial activities [4,5]. Without innovation in technology and processing methods to sustainably meet the demand for clean water, a shortage of clean water is inevitable. Desalination, the process of removing excess salt from water, has proven to be a promising solution for sustainably meeting the demand for clean water. Desalination, typically using seawater, holds great potential due to the abundant availability of seawater and its underutilization.

Currently, most water desalination processes use thermal methods and membrane separation technology[6]. Thermal-based technology involves evaporating seawater and then condensing the resulting vapor to produce clean drinking water. This principle relies on the boiling point difference between pure water and impurities like salt. However, this technology is less effective due to its high operational energy consumption[7]. Consequently, many countries are transitioning to more modern desalination technologies, membrane-based technology. Membrane technology employs separation techniques driven by pressure and concentration gradients. It has gained significant interest in water purification due to its advantages, including good energy efficiency, low operating conditions, and high separation selectivity[8–10]. Currently, the reverse osmosis (RO) process is the most widely used membrane-based desalination process globally. RO is a membrane technology separation method that can separate solutions with low concentrations, such as seawater, from solutions with high concentrations, in this case, clean water[11]. This technology essentially works by applying high pressure exceeding the osmotic pressure on the membrane, causing the flow or diffusion of seawater into freshwater, while salt minerals in the seawater are retained. In the desalination process, the RO process can facilitate particle size filtration down to ≤0.001 µm.

In general, membranes are classified according to their constituent materials into two groups, organic membranes and inorganic membranes. Organic membranes are currently still the main choice on the market and are often used because of their ease of processability, mechanical strength, and excellent material stability [12–15]. Some examples include polyethylene (PE), polytetrafluoroethylene (PTFE), polypropylene (PP), cellulose acetate (CA), and polyvinylidene fluoride (PVDF). Among these examples, PVDF is one of the most frequently used types of polymer membranes for water treatment applications because it has superior salt rejection efficiency (±99%) compared to other types of polymers [16,17]. PVDF is a semi-crystalline polymer, where the crystalline phase provides mechanical strength and resistance to pressure, while the amorphous phase provides flexibility. This semi-crystalline phase is what makes PVDF in demand because of its flexibility and mechanical strength. In addition to its good mechanical stability, PVDF also has stability against corrosive chemicals and organic compounds such as acids and oxidants. However, the main disadvantage of PVDF is its hydrophobic nature. This hydrophobic nature makes PVDF low flux and is susceptible to fouling [18]. Therefore, there has been a lot of research to optimize the use of PVDF as a membrane material by increasing water flux through various modifications to increase hydrophilicity of the PVDF membrane. To enhance the performance of PVDF membranes, fillers such as zeolite Y can be incorporated. Zeolite Y is a microporous aluminosilicate with a high surface area and uniform pore size distribution. Incorporating zeolite Y into PVDF membranes can improve their hydrophilicity, mechanical strength, and rejection rates for salts and other contaminants. The high surface area and unique pore structure of zeolite Y facilitate better water transport while effectively rejecting ions, making it an optimal choice for desalination applications[19–21]. Compared with other zeolites, incorporating zeolite Y into polymeric membranes significantly increased water flux to 141% compared with neat membrane[22].

Preparation of membrane is a crucial step in creating a desalination membrane that is defect free and has low resistance. Formulation of membrane involves a wide range of factors, including filler loading and acid effects. Finding the ideal filler loading in the polymer solution is necessary to provide a thin, defect-free selective layer for the salt desalination process. The degree of entanglement and spread of the polymer chains and compatibility with filler, which is directly connected to the density of the resulting membrane, is determined by the polymer concentration. A thin, porous selective layer is created when a membrane is formed from a mixed matrix membrane which encourages high water flux but low salt rejection. A thicker and denser selective layer, on the other hand, results from better filler loading in the polymer solution, which encourages a higher water flux and salt rejection in this study, PVDF/zeolite Y mixed matrix membranes for salt desalination were prepared by varying zeolite Y loadings in membrane’s casting solution. Zeolite Y variations were carried out in the range of 0.5 - 1.5 wt% to study its effect on membrane’s performance and find optimal fillers in PVDF matrix for desalination application.

**EXPERIMENTAL**

**Materials**

The materials that were used in this study were Polyvinylidene Fluoride (PVDF), Commercial zeolite Y, Deionized Water, n,n-dimethylformamide (DMF, 99.99%, Merck).

**Fabrication of PVDF/Zeolite Y Mixed Matrix Membrane**

Before fabrication of mixed matrix membrane, membrane printing solution was made first. The membrane printing solution was made by dispersing zeolite Y into n,n-dimethylformamide (DMF) according to the desired weight variation. The weight variation of zeolite Y addition in this study was 0.5, 1, and 1.5 wt% which were then designated as MMM-0.5, MMM-1, and MMM-1.5. The zeolite Y dispersion process was carried out with the help of an ultrasonic homogenizer for 60 minutes to ensure that the zeolite Y particles were well dispersed and did not settle. After the mixture of zeolite Y in DMF had been well dispersed, PVDF equivalent to 16 wt% of membrane casting solution was added slowly while stirring at 300 rpm and 60°C. The membrane printing solution was then left under stirring conditions for 24 hours to ensure that the membrane dope was completely homogeneous. Next, the membrane printing solution is left at room temperature for 24 hours for the degassing process of air bubbles trapped during the stirring process. After that, the solution is ready for the fabrication or printing process. The composition of zeolite Y, PVDF, and DMF in each variation of the fabricated membrane has been presented in **Table 1**.

The next process is membrane casting. The membrane solution is poured slowly and evenly on one end of the casting plate and then pulled to the other side using a casting blade. The casting plate is then immediately immersed in a coagulation tank in the form of a plastic tray containing deionized water which acts as non-solvent. This immersion process aims to facilitate the exchange of solvents with non-solvents in the phase inversion process of the NIPS method. After 5 minutes, the membrane has floated and separated from the casting plate completely, indicating that the printed membrane has successfully become a free-standing membrane. The membrane is then transferred into a coagulant containing deionized water for further coagulation and phase inversion processes for 48 hours. After 48 hours, the coagulant water is then replaced with a mixture of deionized water: ethanol 3:1. The process is carried out to ensure that all DMF solvents are completely removed from the membrane. The process of immersing the membrane with a mixture of water and ethanol is carried out 24 hours. Finally, the membrane is then dried at room temperature until completely dry and then stored before being tested for its performance.

**TABLE 1**. Composition of PVDF, zeolite Y, and DMF in Dope Solution

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Type of Membrane** | **PVDF Mass** | **Zeolite Y Mass** | **DMF Mass** | **Total Mass of Solution** |
| **PVDF Neat** | 8 | 0 | 32 | 50 |
| **MMM-0.5** | 8 | 0.04 | 33.56 | 50 |
| **MMM-1** | 8 | 0.08 | 33.52 | 50 |
| **MMM-1.5** | 8 | 0.12 | 33.48 | 50 |

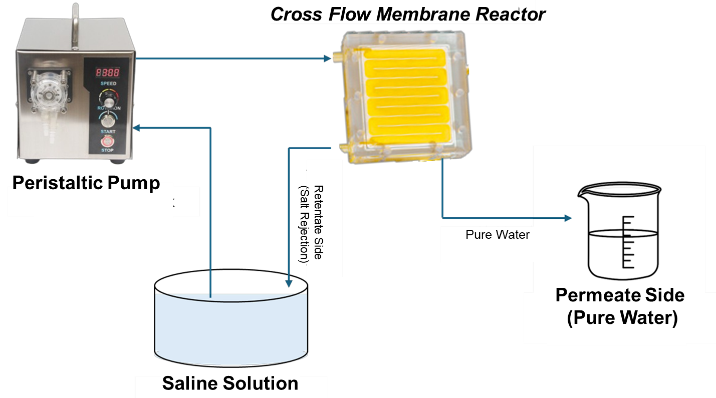
**Characterization**

The crystallinity of commercial Zeolite-Y is identified using X-Ray Diffraction (XRD) wavelength Cu Kα = 1.5406 Å) at 5 to 60° (to obtain a diffractogram in the form of a graph of the relationship between relative intensity and angle 2θ. XRD pattern characterization was carried out using a PANalytical X’Pert PRO instrument using Cu Kα radiation (40 kV, 30 mA). The samples were exposed to X-rays at a scan rate of 2θ = 0.020° s-1. Meanwhile, the hydrophobicity of the membrane is determined by measuring its Water Contact Angle (WCA) using the sessile drop method, which involves placing a 1 μL of water droplet onto the membrane surface. A membrane is classified as hydrophilic when its contact angle is less than 90°.

**Mixed Matrix Membrane Desalination Performance Test**

The performance test of successfully fabricated membrane was conducted to assess effectiveness of the desalination performance of mixed matrix membrane and to determine effect of modification of addition of filler into matrix on membrane performance. In the application of membranes for desalination, main parameters used are measurement of pure water flux and salt rejection. Determination of pure water flux and salt rejection are the main parameters in measuring and assessing the performance of membranes for water desalination. Measurement of pure water flux is carried out by measuring the volume of permeated water that has successfully passed through membrane within a certain period. Conversely, measurement of salt rejection is carried out by calculating salt reduction through measuring the salt concentration in the feed solution and permeate.

where J is the pure air flux (L.m-2.h-1), t is time (h), A is effective membrane surface area (m2), V is permeated volume (L), R is salt rejection (%), Cp is permeated salt concentration (g.L-1), Cf is feed salt concentration (g.L-1). For operational conditions and technical specifications, a peristaltic dosing pump FSD-400 was used to facilitate the flow of brine at a set speed of 100 rpm to achieve the desired pressure (1 bar). The reactor used was Sartorius Vivaflow 50R with a crossflow filtration system and an effective membrane surface area of 0.005 m². Salt concentration measurements were carried out using the CONSTANT Water Quality Tester WT 501 instrument which had been calibrated before testing. The schematic of the test is shown in **Figure 1**.



**FIGURE 1**. Schematic Illustration of Desalination Performance Testing Equipment

**RESULT AND DISCUSSION**

**X-Ray Diffraction Zeolite Y**

Zeolite Y was characterized by using XRD to determine the phase and morphology of zeolite Y formed. The XRD pattern can be seen in **Figure 2**. The results of characterization using XRD were first shown by the XRD diffractogram of zeolite Y with peaks at 2θ = 6.18; 10.07; 11.82; 20.22; 23.48; 26.85; 30.54; 31.17°. In the diffractogram results, there is a peak with the highest intensity at 2θ = 6.18°, which indicates the presence of a zeolite Y crystal plane (111)[13]. The XRD diffractogram results of zeolite Y are in accordance with the previous research, where zeolite Y has a sharp peak with high intensity at 2θ ~6° (111)[23]. XRD diffractogram results of zeolite Y are also in accordance with Powder Diffraction File (PDF) obtained from Joint Committee on Powder Diffraction Standards (JCPDS) Data Base number 39-1380. Thus, it can be concluded that the zeolite Y used is in accordance with Zeolite Y standard and no other phases are formed.

A graph of a graph

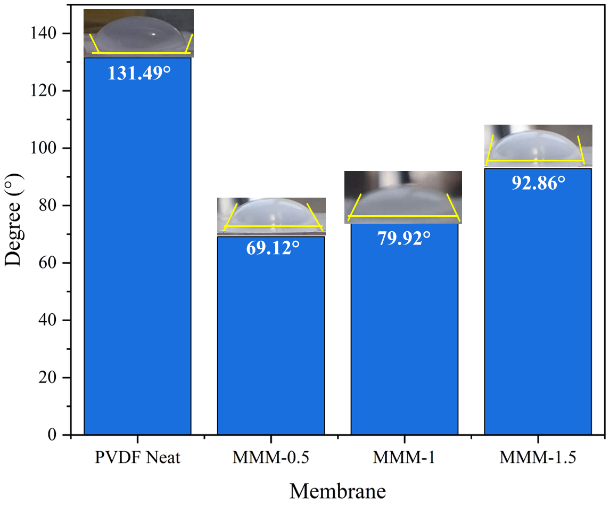
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**FIGURE 2**. XRD Pattern of Zeolite Y

**Water Contact Angle of Membrane**

Water contact angle (WCA) measurement can be seen in **Figure 3**. Based on measurement, it was found that the PVDF Neat membrane had the highest water contact angle, 131.49° (hydrophobic). These results are in line with other studies, where pure polymer membranes generally tend to have hydrophobic properties[24]. This hydrophobic membrane interface makes PVDF Neat polymer membrane have poor wetting properties. Wetting itself refers to the ability of a liquid to make and maintain contact with a membrane surface and the resulting interaction between the two when in contact with each other. The interaction between water and the membrane makes it difficult for water to permeate into the membrane, which may indicate that PVDF Neat membrane has a pure water flux value that is still poor. Upon examining the WCA of PVDF Neat, we find it resonates harmoniously with the inherent properties and architecture of PVDF itself. This alignment is attributed to its non-polar α phase, where arrangement of CH-CF bonds effectively neutralizes the dipole moment within polymer, resulting in its distinctive hydrophobic characteristics. In this membrane, this α phase is still very dominant because it is the most stable phase of the other two phases[25]. The statement of hydrophobic sites of PVDF matrix is aligned with its data and has successfully shown suitability.

Mixed matrix membranes incorporating zeolite Y filler exhibit a decrease of WCA with an increase in filler percentage. The MMM-0.5 membrane transitioned to hydrophilic phase (WCA < 90°). This phenomenon illustrates a significant reduction in WCA for PVDF membranes with zeolite Y relative to neat membranes, achieving hydrophilic behavior. Studies indicate that higher zeolite Y content diminishes the intensity of α-phase peaks in PVDF, which correlates with its non-polar attributes [26]. Reduction in intensity shifts the membrane towards the β-phase, enhancing its polarity and affinity for water, a polar molecule. This observation underscores the physical interaction between PVDF and zeolite Y [27]. The increased membrane polarity, resulting in enhanced affinity for water, renders the mixed matrix membrane hydrophilic, contrasting with the hydrophobic nature of neat PVDF. MMM-0.5 exhibits the lowest water contact angle at 69.12°, aligning with the notion that zeolite Y addition promotes greater polarity and hydrophilicity properties. Consequently, MMM-0.5 is posited to possess superior water flux relative to others. However, the higher percentage of fillers incorporated in membrane matrix, the higher their WCA. Particle agglomeration is the main factor contributing to increased hydrophobicity with higher filler loadings. Excess zeolite-Y loading results in particle clustering near the membrane surface. This agglomeration induces surface roughness that promotes hydrophobicity via the Cassie-Baxter wetting mechanism, which reduces water-surface contact area. Additionally, the hydrophilic contact angle suggests that the membrane exhibits a clogging reduction. The findings confirm that 0.5% zeolite Y filler incorporation effectively enhanced hydrophilic properties and increased reusability.



**FIGURE 3**. Water Contact Angle on Mixed Matrix Membrane

**Water Flux and Salt Rejection Performance of Membrane**

All measurements of water flux and salt rejection are presented in **Table 2**. The PVDF membrane exhibited the lowest water flux at 26.07 L.m-2.h-1. Conversely, MMM-0.5 recorded the highest pure water flux at 217.91 L.m-2.h-1. A trend is observed where increasing zeolite Y filler mass percentage correlates with enhanced pure water flux. This water contact angle measurements and membrane characterization data are interconnected. The increase in membrane polarity enhanced water affinity in the membrane surface. This trend supports the observed increase in pure water flux with higher zeolite Y filler percentages. The significant 736% increase in pure water flux at MMM-0.5 compared to PVDF Neat is noteworthy. The increase in flux is attributed to enhanced polarity as indicated by water contact angle measurements. The incorporation of higher zeolite Y (MMM-1 and MMM-1.5) indicated reduction in water flux. One plausible explanation for this membrane performance is that the water contact angle increases and is near to hydrophobic properties. At 1.5% zeolite-Y, the performance slightly increased. This observation is notable because, at 1.5 wt% zeolite-Y, the network reorganization and porosity mitigate agglomeration. Despite heightened surface roughness and tortuosity, the increased zeolite sites and void interconnectivity lead to a flux recovery of 183.79 L·m⁻²·h⁻¹. In this context, the membrane attains a state where increased active surface area and hydrophilicity enhance transport, although it is constrained by mass-transfer resistance from filler clustering. The maximum flux at low loading (0.5%) indicates optimal dispersion, whereas the decline and partial recovery at elevated loadings result from zeolite-induced pore structure enhancement and agglomeration.

The non-monotonic relationship in salt rejection and zeolite Y loading results from the interplay between enhanced selective sites at lower filler levels and structural disruption at increased loadings. At 0.5 wt% zeolite Y, the zeolite crystals generate hydrophilic and adsorption sites, improving water transport and increasing salt rejection from 70.26% to 73.38% through enhanced selectivity and minimal structural damage. At 1 wt% zeolite Y, the effects are optimized. The extensive zeolite network enhances water pathways while preserving membrane integrity, further elevating rejection percentage by amplifying water flux and selectivity through stronger affinity differentials between water and salt [19]. However, at 1.5 wt% zeolite Y, filler agglomeration and overloading disrupt the dense selective layer and supporting structure, leading to particle clusters that obstruct pores and reduce the uniformity, thereby facilitating salt permeation. As a result, rejection declines significantly to 62.61%, as the increased zeolite content cannot compensate for the enhanced mass-transfer resistance and the emergence of nonselective pores [17].

**TABLE 2**. Water Flux and Salt Rejection Performance of Membrane

|  |  |  |
| --- | --- | --- |
| **Type of Membrane** | **Water Flux (L.m-2.h-1)** | **Salt Rejection (%)** |
| **PVDF Neat** | 26.07 | 70.26 |
| **MMM-0.5** | 217.91 | 73.38 |
| **MMM-1** | 173.68 | 79.32 |
| **MMM-1.5** | 183.79 | 62.61 |

# CONCLUSION

The incorporation of zeolite Y into the PVDF matrix significantly improves the membrane’s performance at optimal loading concentrations. At 0.5 wt% zeolite Y, there was a notable increase in air flux alongside a minor rise in salt concentration. 1 wt% zeolite-Y sustained elevated flux levels while optimizing salt rejection, thus exhibiting considerable advancements compared to the neat membrane. This superior performance was ascribed to the homogeneous distribution of zeolite crystals, which improved hydrophilicity and adsorption without forming a resonance layer. Conversely, at a concentration of 1.5 wt%, the performance declined due to agglomeration and obstruction, leading to diminished salt rejection and flux reduction. These results suggest that a 1 wt% loading of zeolite Y represents the most advantageous compromise for water desalination applications involving mixed matrices. Future work could explore further optimization conditions for MMMs, for example, by investigating zeolite activation method to search for MMMs that provide stable performance over time.

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