

Production of desalination membranes from recycled PVDF polymers

A. Katırcı^{1*}, F. U. Nigiz²

¹Çanakkale Onsekiz Mart University, School of Graduate Studies, Çanakkale, 17100, Türkiye

²Çanakkale Onsekiz Mart University, Department of Chemical Engineering, Çanakkale, 17100, Türkiye

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Water is of critical importance to industry. Numerous technologies have been developed regarding water use in industrial applications. In this study an innovative membrane distillation method is investigated for the separation of brackish water that can be used in process and cooling water applications. The primary focus of this study is the use of a membrane recovered from waste polymers in a membrane distillation system. This approach will prevent waste from entering the environment by facilitating its revaluation, while also contributing to the national economy by promoting recycling as an alternative to the use of new products. To this end, polyvinylidene fluoride (PVDF), a commercial membrane material, was recycled from waste and reused. The recovered PVDF was converted into a porous membrane structure using the electrospinning method. The membrane surface was modified using poly(vinyl alcohol) (PVA) via the dip-coating method. Characterization of the produced membranes included FTIR, DSC, and mechanical strength analyses. Salt separation efficiencies exceeding 99% were achieved in membrane performance evaluations.

Keywords: Desalination, Membrane distillation, PVDF, Recycling

INTRODUCTION

Industrial water use is a critical area of study and research that is growing day by day alongside technological and industrial advancements, yet due to environmental concerns, it is essential to ensure that water is reused as much as possible. In industrial processes, water is used as process water and cooling water. In particular, facilities with high water consumption (such as thermal power plants) are located near natural water sources and treat saltwater for use in their systems. Desalination, literally meaning “removal of salt,” can be described as a technological process of obtaining fresh drinking water from brackish or saltwater. Thanks to desalination technology, seawater resources are utilized to produce drinking water, providing a solution to water scarcity in the face of a growing population. It also supports the sustainability of agriculture and industry. There are two primary technological approaches for removing salt from water: thermal and membrane-based methods.

Membrane distillation is a desalination method used to convert saltwater into potable water. In this system which primarily uses hydrophobic and porous membranes, the separation gradient is driven by both temperature and partial pressure differences. As water vapor generated by heat in the feed section passes through the hydrophobic membrane, salt and contaminants are separated. Membrane distillation is a method that operates at low temperatures, requires

no pretreatment, and utilizes waste heat as a heat source. From this perspective, it offers significant economic and environmental advantages [1].

Vacuum membrane distillation (VMD) is one of the membrane distillation techniques. It is used in desalination processes to separate salt and other dissolved substances from seawater. This distillation technique is based on the principle of conducting the evaporation process under vacuum. Applying a vacuum to create low pressure lowers the boiling point, allowing the system to operate at lower temperatures. As a result, less energy is consumed compared to other distillation techniques, since the energy required to reach high temperatures is minimized. In the VMD process, the saltwater is heated and fed to one side of the membrane under vacuum. The heated water evaporates and passes through the semi-permeable membrane, while the salt and impurities remain behind. The vapor is cooled in the vacuum environment and condenses, resulting in the production of pure, salt-free water. High salt removal is achieved [2]. The most important component of this system is the membrane used.

There are several key characteristics that determine the performance of membranes used in desalination processes. A high water flux is critical for reducing process time and lowering energy costs. Low fouling tendency contributes to reduced maintenance requirements and extended service life for the membrane. Additionally, low energy requirements enable high efficiency to be achieved

* To whom all correspondence should be sent:

E-mail: katirciaysenur@gmail.com

with minimal energy input. Mechanical durability ensures physical integrity of the membrane over an extended period, while high salt rejection capacity allows for the maximum extraction of pure water from saline water. Thermal stability supports the membrane's ability to operate at high temperatures without structural degradation. Furthermore, high porosity facilitates the passage of water vapor while limiting the passage of dissolved substances [1].

Recycling reduces dependence on raw materials required for new polymer production. The methods used to dispose of membranes—landfilling and incineration—are not environmentally sound. For these reasons, new methods for disposing of these membranes are needed. It has been demonstrated that recycled polymers reduce both operational costs and carbon emissions. Processes that continue without the need to constantly replace membranes by using recycled polymer membranes are highly beneficial to the operation in terms of cost. Moreover, since the polymers do not need to be replaced continuously, there is no waste problem, and because no energy is consumed, they are environmentally friendly. Furthermore, the pollution problem caused by the membranes used is eliminated, and thanks to recycled polymer membranes, there is no longer a need for pollution removal processes.

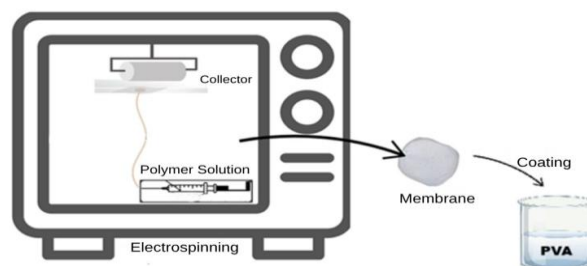
The literature contains numerous significant studies on the use of membrane distillation systems to produce pure water from seawater. The majority of these studies have been conducted using polyvinylidene fluoride (PVDF). PVDF is an important polymer that is non-reactive, thermally stable, and mechanically strong, with good processability and chemical resistance to various materials such as acids, bases, organic solvents, and oils [3, 4]. It is a microfiltration polymer and is commercially available. However, particularly in water treatment processes, its hydrophobic nature makes it prone to membrane fouling; to address this and enhance its hydrophilicity, various modifications can be applied. PVDF membranes are widely used in various fields such as microfiltration, ultrafiltration, membrane bioreactors, membrane distillation, gas separation, and stripping [5-7].

In recent years, it has been observed that PVDF is recyclable, just like polyethylene terephthalate, polystyrene, and polyamide, and that it retains its properties after recycling. PVDF was included in this project specifically because it serves as the primary material for microfiltration membranes and because of the concept that these membranes can be recycled and reused after use. New studies on recycled PVDF have begun to emerge in the

literature [8]. Therefore, within the scope of this project, the aim is to produce purified water *via* membrane distillation using recycled PVDF produced by the electrospinning method.

EXPERIMENTAL

A DMF-acetone solution containing PVDF (20% by weight) and PVP (10% by weight relative to PVDF) was prepared and stirred at 50 °C. The homogeneous solution was drawn into a syringe and loaded into an electrospinning system. The solution was collected onto a plate at a voltage of 17 kV, a flow rate of 0.05 mL/min, and a distance of 150 mm. Subsequently, the produced PVDF membranes were modified using a dip-coating technique. The membranes were immersed in a 10% (w/w) aqueous polyvinyl alcohol (PVA) solution for a specific duration, followed by a drying process to ensure the formation of a thin, homogeneous PVA layer on the nanofiber surfaces. This modification step was used to optimize the membrane's desalination performance. The samples were coded according to the number of coating cycles; each cycle contributed approximately 2 µm to the total coating thickness. Scheme 1 illustrates the electrospinning and PVA coating method used in membrane production.



Scheme 1. Schematic representation of the electrospinning and PVA coating methods in membrane production

FTIR spectra were recorded on a Shimadzu Tracer spectrometer (IRTracer-100, Shimadzu Corporation, Kyoto, Japan). A differential scanning calorimeter (DSC-60 Plus, Shimadzu Corporation, Kyoto, Japan) was used for DSC analysis. In compliance with the ASTM D882 standard, the tensile strength and elongation at break values were measured during the mechanical testing phase using the ANKARIN universal testing machine. The agar diffusion method was used to conduct antimicrobial tests [9]. The test microorganisms were *Staphylococcus aureus* (+) and *Escherichia coli* (-), which were seeded separately on liquid media and maintained at 37°C for one day.

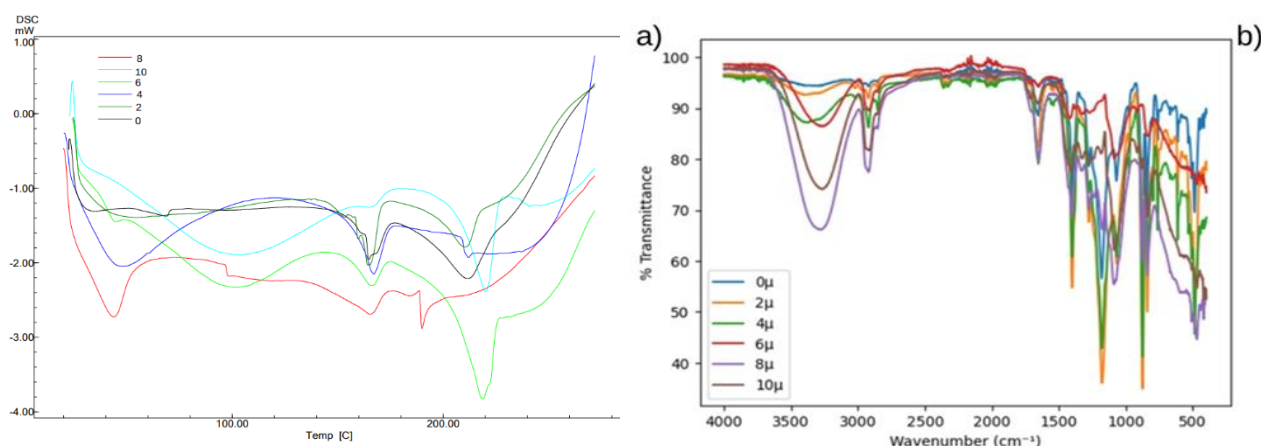


Figure 1. FTIR spectra (a) and DSC analysis (b) of recycled PVDF-PVA coating membranes

After reducing it to 0.5 McFarland turbidity, a sterile brush was used to evenly disperse it across the agar surface. Film samples were placed on Petri dishes and stored at 37°C for one day. In the end, the diameter of the formed zones was measured.

RESULTS AND DISCUSSION

Characterization tests

As part of the characterization tests, FTIR, DSC, mechanical analysis, and contact angle tests were performed. The disk diffusion method was used for antimicrobial analysis.

- **FTIR.** The FTIR spectra are shown in Figure 1a. The FTIR analysis confirmed that the PVA modification has been successfully integrated into the PVDF membrane surface. The broad peaks observed around 3300 cm^{-1} in all spectra confirm the presence of hydroxyl (-OH) groups in the PVA structure [10, 11]. The fact that the peak's intensity is highest in the 8 μm - and 10 μm -samples indicates that the most dense coating occurred in these samples. On the other hand, the decrease in the intensity of the -OH peak in the 2 μm and 0 μm samples indicates a reduction in coating thickness.

- **DSC.** The DSC analysis results are presented in Figure 1b. The broad endothermic peaks observed between 50 and 100°C indicate the removal of moisture from the structure. Additionally, the peaks between 40 and 70 °C can be associated with the glass transition temperature of PVA [11]. The fact that these peaks are more pronounced at higher coating thicknesses (8–10 μm) indicates an increase in PVA content. The sharp melting peaks around 170°C and 220°C represent the melting temperature (T_m) of PVA's crystalline regions [11]. The weaker presence of this effect at 2 μm and 0 μm indicates a low coating amount and a low PVA content. In the 6 μm sample, a deep endothermic peak (~220 °C) indicates a regular structure and optimal PVA distribution. Since the classic melting peak of PVDF

is not very clear, the PVA coating may have masked the crystallization of PVDF.

- **Mechanical analysis.** The results of the mechanical analysis are presented in Table 1. Among the tested samples, the 6 μm -sample exhibited superior mechanical strength with a peak tensile stress of approximately 31 MPa, indicating an optimal balance between the recycled PVDF matrix and the modification process. Although the 10 μm -sample exhibited the highest flexibility with an elongation value exceeding 0.40, its lower tensile capacity indicates a structure that is more ductile but less resistant to pressure. A consistent decrease in both mechanical strength and flexibility was observed from 4 μm down to 0 μm .

Table 1. Results of the mechanical analysis

Membrane	Stress (MPa)	Strain
10 μ	12.29	0.41
8 μ	21.19	0.22
6 μ	31.15	0.31
4 μ	9.59	0.28
2 μ	8.13	0.25
0 μ	5.89	0.18

Contact angle analysis

The contact angle results are presented in Figure 2. As the PVA coating thickness increased, the contact angle showed a significant decrease from 76° to 44°, indicating a significant increase in surface hydrophilicity. This is attributed to the increased presence of PVA-derived hydroxyl (-OH) functional groups, which support hydrogen bonding with water molecules. A more pronounced decrease beyond 6 μm indicates the formation of a more homogeneous and continuous coating layer. These findings confirm that PVA modification effectively improves the wettability of PVDF membranes.

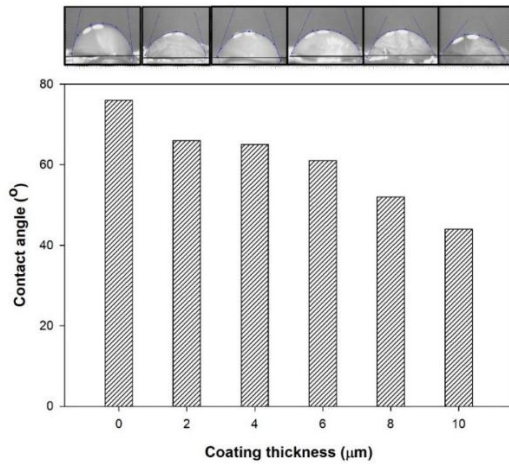


Figure 2. Contact angle results for recycled PVDF-PVA-coated membranes

Antimicrobial analysis

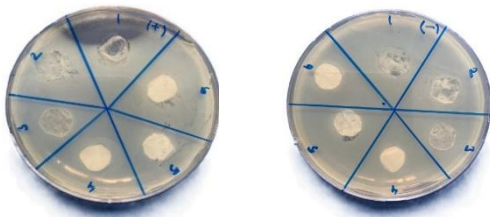


Figure 3. Antimicrobial analysis results for recycled PVDF-PVA coating membranes

The results of the antimicrobial tests conducted by the disk diffusion method are presented in Figure 3. No distinct zone of inhibition is seen around the samples. In this case, there is no strong antibacterial activity. Since PVA is not an antimicrobial substance

by nature, this result is expected [12]. Consequently, the modification of PVA has not imparted antibacterial properties to the PVDF membranes.

Desalination tests

The graph in Figure 4a shows that the flux value gradually decreases as the coating thickness increases from 0 μm to 10 μm . The flux, which was approximately 11.6 $\text{kg}/\text{m}^2\text{h}$ at the start (~ 0 μm), has decreased to approximately 6.4 $\text{kg}/\text{m}^2\text{h}$ at a thickness of 10 μm . As the coating layer thickens, the mass transfer resistance that water molecules must overcome, increases. This is directly proportional to Fick's law; the increase in resistance reduces the effect of the driving force, thereby lowering permeability. It is observed that salt retention efficiency dramatically increases as coating thickness increases. While the rejection rate was below 30% in the uncoated (0 μm) state, even a 2 μm coating alone raised this rate to over 70%. At thicknesses of 8 μm and 10 μm , the system has nearly reached a 100% (full efficiency) rejection rate. The coating layer seals defects and large pores in the membrane, allowing the dissolution-diffusion mechanism to become dominant. This permits only water molecules to pass through while blocking salt ions. The critical range in the graph is between 4 μm and 6 μm . This is because beyond 8 μm , the salt rejection rate has already reached saturation (very close to 100%), yet the flux continues to decrease. Therefore, it can be concluded that this thickness is the most suitable.

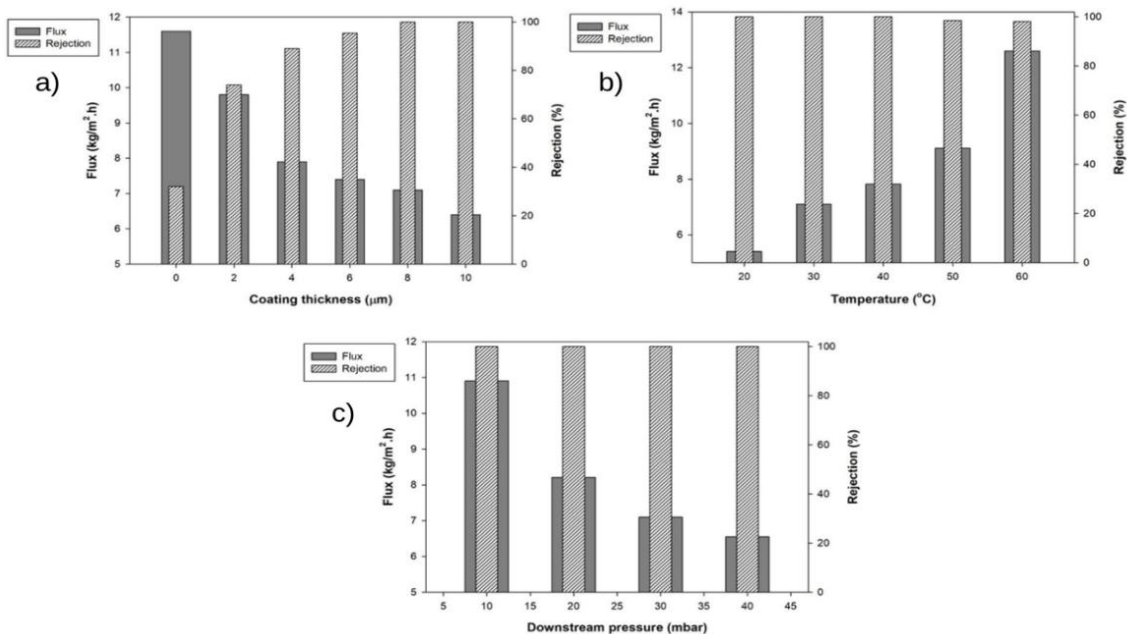


Figure 4. Effect of operating parameters on VMD performance: coating thickness vs. permeate flux and salt rejection (a), feed temperature vs. permeate flux and salt rejection (b) and downstream pressure vs. permeate flux and salt rejection (c).

The effect of temperature is one of the most dynamic parameters in membrane processes, both from a thermodynamic and kinetic perspective. When the temperature rose from 20°C to 60°C, the flux value increased from 5.4 to 12.6 kg/m²h, representing an increase of over 130% (Figure 4b). This is an expected phenomenon and has two main causes: as water temperature increases, its viscosity decreases. The thermal motion of the polymer chains in the membrane increases with temperature. This creates temporary free volumes within the polymer, thereby increasing the diffusion rate of water. This relationship is typically explained by the Arrhenius equation: an arithmetic increase in temperature leads to a logarithmic/exponential increase in flux.

As temperature increases, the kinetic energy of water molecules increases directly. This affects the transport within the membrane as follows:

- An increase in temperature enhances the thermal vibrations of water molecules, causing them to “bounce” more rapidly through the voids in the membrane’s polymeric matrix.
- According to the Stokes-Einstein equation, the diffusion coefficient is directly proportional to temperature and inversely proportional to viscosity. Thus, as temperature rises, the diffusion coefficient increases, which is the primary cause of the flow increase.

The rise in temperature triggers the primary driving force—namely, the vapor pressure difference. According to Antoine’s equation, as the temperature on the feed side increases, the vapor pressure of the liquid exponentially increases. The partial pressure difference between the feed side (hot) and the product side (vacuum) of the membrane sharply increases. The flow rate is directly proportional to this pressure difference.

Figure 4b shows that salt rejection performance remains remarkably stable at 99.99% up to 40°C, but drops slightly to 98.1% at 50°C and 60°C. The structural integrity of the membrane is very well preserved in the 20–40°C range. The membrane successfully prevented the passage of salt ions despite the increase in flux. At 50–60°C, the slight decrease in salt rejection is due to the accelerated diffusion of salt ions. Like water molecules, the kinetic energy of salt ions also increases. Additionally, excessive relaxation of polymer chains at high temperatures may create microscopic pathways through which salt ions can pass to widen.

In the graph in Figure 4c, we see that the flux value decreases as the feed pressure increases from

10 mbar to 40 mbar (from 10.90 to 6.55 kg/m²·h). In pervaporation, the driving force is the difference in partial vapor pressure on either side of the membrane. The lower the pressure on the feed side, the stronger is the force drawing water molecules to the other side. When the feed pressure increases from 10 to 40 mbar, the vacuum force decreases. This increases the water vapor concentration on the product side of the membrane, thereby weakening the driving force. As a result, the flux decreases.

The most striking performance metric regarding pressure is that salt rejection remains constant at 99.99%, regardless of the vacuum pressure. This is due to the fact that salt is non-volatile. In pervaporation and membrane distillation, as water passes through the membrane and crosses to the other side in the vapor phase, salt ions remain in the liquid phase and cannot evaporate. As long as changes in vacuum pressure do not disrupt the pore structure of the membrane’s selective layer, salt permeation theoretically remains close to zero. The highest flow rate was achieved at 10 mbar. This is the most efficient point from an industrial perspective because the highest product yield per unit time was obtained at this pressure. The lowest flow rate was achieved at 40 mbar. While this can reduce the energy consumption of the vacuum pump, it significantly lowers production capacity.

The graph in Figure 5 shows that the flux value starts at approximately 7.1 kg/m²·h in the first experiment and decreases at a slight downward slope to approximately 6.7 kg/m²·h by the end of the 10th experiment. This decrease of approximately 5–6% is considered quite acceptable in the membrane literature. Salt ions accumulating on the membrane surface over time may slightly impede water passage. Under continuous pressure or vacuum, the polymer chains may adopt a tighter packing arrangement, which could slightly reduce the flux. If the feed water contains trace amounts of organic matter, these may slightly block the pores/free volumes in the active region.

The most impressive aspect here is that the salt rejection curve remains at the 100% (or 99.99%) line. The fact that selectivity remained completely intact over 10 cycles demonstrates that the active layer adheres very well to the substrate and does not experience mechanical issues such as peeling, cracking, or swelling during operation. This stability is also a strong indication that the membrane can return to its original performance after cleaning cycles.

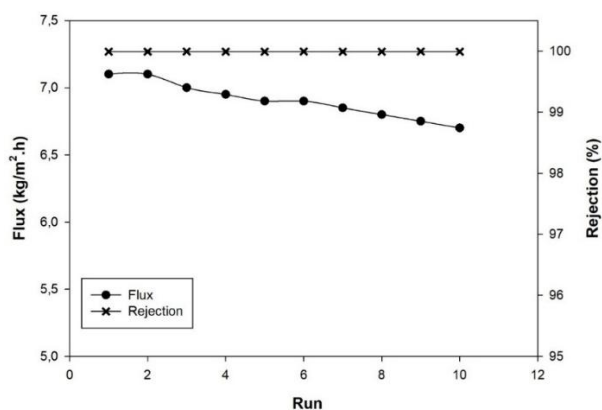


Figure 5. Evaluation of the membrane's reusability performance based on flux and rejection efficiency

CONCLUSIONS

This study has demonstrated that PVDF polymer recycled from waste membranes can be converted into high-performance desalination membranes by the electrospinning technique. Characterization studies confirmed that the PVA coating was successfully integrated into the membrane surface and that as the coating thickness increased, the surface acquired a more hydrophilic structure (with the contact angle decreasing from 76° to 44°). In desalination tests, it was observed that an increase in temperature raised the flux value by over 130% in accordance with the Arrhenius equation, while vacuum pressure directly affected the filtration rate by triggering the driving force. In particular, achieving a salt rejection rate of 99.99% at a coating thickness of 8 µm and above demonstrates the commercial potential of the domestically produced membrane. The fact that selectivity was maintained and flux loss remained at an acceptable level of 6% throughout 10 cycles of stability testing supports the membrane's long-term usability in industrial processes. In conclusion, this project has contributed to both the prevention of environmental pollution through the recovery of waste polymers and the development of a sustainable desalination technology with high economic value.

Abbreviations:

DSC: Differential scanning calorimeter

FTIR: Fourier transform infrared spectroscopy

VMD: Vacuum membrane distillation

PVA: Polyvinyl alcohol

PVDF: Polyvinylidene fluoride

Declaration of competing interest: The authors affirm the absence of any known competing financial

interests or personal relationships that may have influenced the work presented in this paper.

Author contribution statement: Ayşenur Katırcı: investigation, writing - original draft, carried out the experiment. Filiz Uğur Nigiz: investigation, visualization, conceptualization, methodology, writing - original draft, methodology, data curation, validation, writing - review & editing.

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