THERMAL ANNEALING - INDUCED CHANGES IN POLYETHERSULFONE (PES) MEMBRANES: STRUCTURAL, MECHANICAL, AND FLUX CHARACTERIZATION

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ABSTRACT

Polyethersulfone (PES) membranes are known for their outstanding chemical and thermal resistance, making them suitable for various separation processes. However, enhancement in mechanical properties anremaination efficiency remains necessary for achieving optimal membrane functionality. This study investigates the influence of thermal annealing treatment at low temperature of 60°C and 70°C on the morphology, chemical structure, mechanical performance, and pure water flux of PES membranes, compared to none - thermal annealing membranes. Commercial membranes were subjected to thermal treatment for 60 s using a laboratory oven. Characterization techniques included Fourier Transform Infrared Spectroscopy (FT - IR) for functional group analysis, Scanning Electron Microscopy (SEM) for morphological assessment, tensile testing for mechanical evaluation, and dead - end filtration for pure water flux measurement. None - thermal annealing membranes exhibited a tensile strength of 7 MPa, elongation of 43 %, and a water flux of 100 LMH. Annealing at 60°C enhanced pore compactness and uniformity, increasing tensile strength to 9 MPa, elongation to 50 %, and flux to 125 LMH. In contrast, treatment at 70°C further increased water flux to approximately 300 LMH but reduced tensile strength to 6 MPa and elongation to 37 %, indicating a more rigid and brittle structure. FT - IR analysis confirmed no chemical degradation, with enhanced hydrophilic group intensities suggesting improved molecular orientation due to the thermal treatment. The results demonstrate that thermal annealing, particularly at 60°C, can effectively improve PES membrane performance relative to none - thermal annealing membranes while preserving structural integrity, offering a practical approach for advanced membrane - based separation technologies.

Keywords: low temperature, mechanical strength, thermal annealing, polyethersulfone membrane, water flux.

INTRODUCTION

Organic polymer - based membranes play a crucial role in water and wastewater treatment due to their affordability, flexibility, and adaptability [1 - 6]. Polymeric membranes are widely used in filtration processes like ultrafiltration, nanofiltration, and reverse osmosis to separate contaminants, salts, and particles

from water [1, 7, 8]. One of the main advantages of polymeric membranes is their ability to be customized to meet specific needs. By modifying properties such as hydrophilicity, mechanical strength, and chemical resistance, these membranes can be tailored for a variety of water treatment applications [9, 10]. For instance, PES membranes offer high filtration efficiency at a lower cost compared to inorganic membranes. Additionally, PES membranes are known for their good resistance to fouling, making them ideal for applications for heavy metal removal [11], organic and oily wastewater [12 - 15], industrial dyes [10, 16, 17] and desalination [18].

Modification of PES membranes has a significant impact on separation performance, including enhanced thermal stability, chemical resistance, and mechanical strength, making PES a promising material for separation applications [19]. Various modification strategies have been explored, such as the incorporation of nanoparticles like silica-based [20], carbon-based [11, 21], graphene oxide [23] and metal organic framework [24, 25] to improve hydrophobicity and reduce fouling. Other methods include chemical grafting and interfacial polymerization [26]. In addition, to modifying the base material and casting techniques, post-production treatments such as thermal annealing have also been investigated. Thermal annealing involves heating the membrane at a controlled temperature below its melting point to improve polymer chain ordering, relieve internal stresses, and stabilize shape and structure of membrane. This treatment is believed to enhance membrane permeability by altering pore size distribution and surface morphology [27].

Thermal annealing works by reorganizing polymer chains at elevated temperatures, which can increase structural density, regulate pore distribution, and strengthen intermolecular bonding [28]. Many previous studies have focused on high - temperature thermal annealing, approaching or exceeding the glass transition temperature (Tg) of PES (220°C) [19]. Under these conditions, increased material density and stiffness can improve mechanical strength but may reduce membrane flexibility and permeability [29]. However, the effects of low thermal annealing at temperatures well below Tg which remains underexplored, particularly for unmodified membranes. This low treatment is promising as it may improve structural performance without risking material degradation or thermal damage. From an industrial perspective, lower annealing temperatures are also more energy - efficient and safer for large-scale implementation [30].

In polymer membranes, thermal annealing plays a crucial role in polymer reconstruction and improved hydrophilicity. Heating the membrane at specific temperatures causes rearrangement of polymer chains within the membrane structure. When heated near Tg, polymer chains gain mobility and can reorganize to form a more ordered structure [31]. This reconstruction enhances the polymer density within the membrane matrix, improving its structural stability, mechanical strength, and thermal durability. It also helps eliminate surface roughness or inhomogeneities that could affect membrane performance. Such molecular - level rearrangement can reduce irregular pores and yield a more uniform pore distribution, improving separation efficiency and filtration performance [32].

In membrane research, characterization - based approaches are essential to understanding how specific treatments influence the resulting physical and functional membrane properties. Structural characterization techniques such as infrared spectroscopy (FT-IR), mechanical testing, and permeability evaluations are widely used to assess changes in microstructure, surface chemistry, and filtration performance. These analyses form the basis for optimizing production processes and tailoring membrane properties for specific applications. Characterization in this study includes scanning electron microscopy (SEM) for morphological assessment, FT - IR for functional group analysis, tensile testing for mechanical properties, and pure water flux testing for filtration performance [33].

This study is particularly important as it offers a simple yet effective approach to improving PES membrane characteristics through thermal treatment. By merely adjusting the post - production temperature, significant changes in structural and functional properties can be achieved without introducing new materials. This supports the sustainability of production processes and is economically and practically beneficial for industry. Thermal annealing - based optimization offers a promising path toward developing more efficient membranes for sustainable industrial wastewater treatment. The objective of this study is to evaluate the impact of thermal annealing on the structural and mechanical characteristics of PES membranes. Through

a comprehensive characterization approach, the study aims to contribute to the development of more reliable and application - oriented membrane designs.

EXPERIMENTAL

Materials

The membrane used in this study was a commercial flat sheet PES membrane with a thickness of approximately 200 μm. The membrane was cut into circular specimens according to the effective filtration area of the dead - end filtration cell. Prior to thermal annealing and filtration testing, the membrane samples were rinsed with distilled water to remove surface impurities and then dried at room temperature.

Thermal annealing treatment

The thermal annealing temperatures were selected as mild post - treatment conditions to improve the physical stability of the PES membrane without causing severe deformation or thermal degradation. PES is known as a thermally stable polymer with a high glass transition temperature of approximately 220 - 225°C; therefore, annealing at 60°C and 70°C was intentionally performed far below its glass transition temperature to maintain the chemical integrity of the membrane while allowing limited polymer chain relaxation and structural stabilization [13, 27]. The use of low - temperature annealing was also intended to minimize pore collapse while evaluating whether mild thermal exposure could influence membrane morphology, mechanical strength, and water flux.

The oven was first set at 60°C. After the set temperature was reached, the PES membrane was placed in the oven for 60 s. The same procedure was then repeated at 70°C. The annealing time was kept constants for all samples to specifically evaluate the effect of annealing temperature.

Membrane characterization

Chemical structure

The chemical structure was observed using Thermo Scientific Nicolet iS10 FT - IR spectrometer (Waltham, MA, USA).

Morphology of membrane

The surface and cross - section of membranes were observed by using scanning electron microscope (JSM6510LA, Tokyo, Japan). All measurements were performed at 15 kV.

Determination of membrane tensile properties

Tensile properties test was conducted using Tensile Testing Machine M1Type, Cometech Testing Machine. The specimen size of membrane was 10 mm x 50 mm and thickness of 200 mm. The test was following ASTM D882, standard tensile properties test for thin plastics sheeting thinner than 1 mm in thickness, with crosshead speed of 10 mm/min and gauge length of 30 mm. Three specimens were tested for every point of data.

Pure water flux calculation

Pure water flux was measured using a laboratory scale dead - end filtration unit equipped with a membrane holder and an effective membrane filtration area of 0.0017 m². Distilled water was used as the feed solution. The filtration experiments were conducted under a constant vacuum pressure of 0.3 bar, which was maintained throughout the testing period. No pressure variation was applied during the experiments, ensuring that any differences in flux were primarily associated with changes in membrane properties resulting from the thermal annealing treatment. The permeate volume was collected over a fixed filtration time, and the pure water flux was calculated using Eq. (1):

$$F_p = \frac{V_p}{A \times \Delta t} \quad (1)$$

where Fp, Vp, A and Δt represent pure water flux, (LMH); permeate volume collected during filtration time, (L); the effective membrane area, (m²); and filtration time (h), respectively.

RESULTS AND DISCUSSION

The annealing treatment applied in this study should be regarded as a low - temperature or mild thermal post - treatment rather than high - temperature annealing near the glass transition region of PES. The temperatures of 60°C and 70°C were selected to avoid thermal degradation, severe pore shrinkage, or collapse of the membrane structure, while still allowing limited physical relaxation of the polymer chains. Previous studies have reported that thermal annealing may influence

membrane morphology, free volume, pore structure, and separation performance depending on the applied temperature and duration [27, 31, 32, 41]. However, because the temperatures used in this study were far below the glass transition temperature of PES, the expected changes were limited to physical stabilization and minor structural rearrangement rather than major chemical or morphological transformation.

The annealing time was fixed at 60 s for all samples to isolate the effect of annealing temperature and to prevent the introduction of an additional experimental variable. Therefore, the influence of annealing duration on membrane structure was not systematically investigated in the present study. This limitation should be acknowledged because longer annealing times may produce different effects on pore structure, polymer chain packing, and membrane performance. Accordingly, the results of this study are interpreted cautiously, and the observed changes in flux and mechanical properties are attributed to mild physical rearrangement induced by low - temperature thermal treatment rather than substantial modification of the PES chemical structure.

From a surface chemistry perspective of PES membrane, thermal annealing enhances the interaction among hydrophilic functional groups such as sulfonate (- SO_3H) and hydroxyl (- OH) [25, 29]. The improved orientation and exposure of these functional groups strengthen the hydrophilicity of membrane, facilitating water molecule adsorption and reducing the affinity for hydrophobic compounds that cause fouling. Additionally, thermal annealing aids in the evaporation of residual solvents or volatile compounds, resulting in a cleaner and more chemically active membrane surface [27].

Mechanically, the increased structural ordering from thermal annealing contributes to improved tensile strength and elongation at break. Stronger and more organized intermolecular bonds provide better structural stability, enabling the membrane to withstand high operational pressures without compromising its physical integrity. This is particularly important for maintaining long - term membrane performance in filtration applications [34].

In terms of separation performance, the enhancement in hydrophilicity and improved pore control allows for higher pure water flux. Water can flow more efficiently through well - organized channels and a more hydrated surface, reducing flow resistance and increasing process efficiency [35]. Furthermore, membrane selectivity for molecules based on size or polarity can also be improved by the denser and more homogeneous polymer structure resulting from thermal annealing [36].

The effect of thermal annealing on membrane morphology

Thermal annealing induces significant changes in the microstructure of membrane, primarily by enhancing the structural regularity of the polymer matrix. This leads to a reduction in pore size irregularities, resulting in more organized and uniform pores, although slightly larger compared to those in none - thermal annealing membranes [32]. As shown in Fig. 1, SEM images reveal that membrane A (none - thermal annealing) exhibits smaller, less regular pores with limited interconnectivity. In contrast, membranes B and C, which were thermally annealed at 60°C and 70°C respectively, display larger, more uniform, and well -connected pore structures. Polypropylene annealed at 50 - 200°C demonstrated the influence of annealing temperature on recrystallization behaviour and supported the “core - shell sandwich” structural model, which provides an explanation for the microstructural evolution of the precursor film during annealing and the development of pores in the samples upon stretching [37].

These changes are attributed to the redistribution and rearrangement of polymer chains during heating, which promotes structural ordering and influences pore distribution within the membrane [29]. The outcome is a decrease in flow resistance and an increase in water flux, despite the enlarged pore size. Thermal annealing can also increase the viscosity of residual solvent, thereby affecting the pore formation dynamics [26]. At higher temperatures (as in Membrane C), the localized evaporation rate of solvent is reduced, promoting the formation of larger yet more interconnected pores.

Membrane A exhibits a denser pore structure, likely due to faster drying without thermal treatment, resulting in more compressed and closed pores. In contrast, thermal annealing slows down the drying process, allowing sufficient time for pore structure rearrangement, leading to a more open configuration. The pore sizes in membranes B and C are nearly identical. The increase of 10°C in annealing temperature, which remains well below the glass transition temperature (Tg) of PES, does not cause a significant difference in pore size. However,

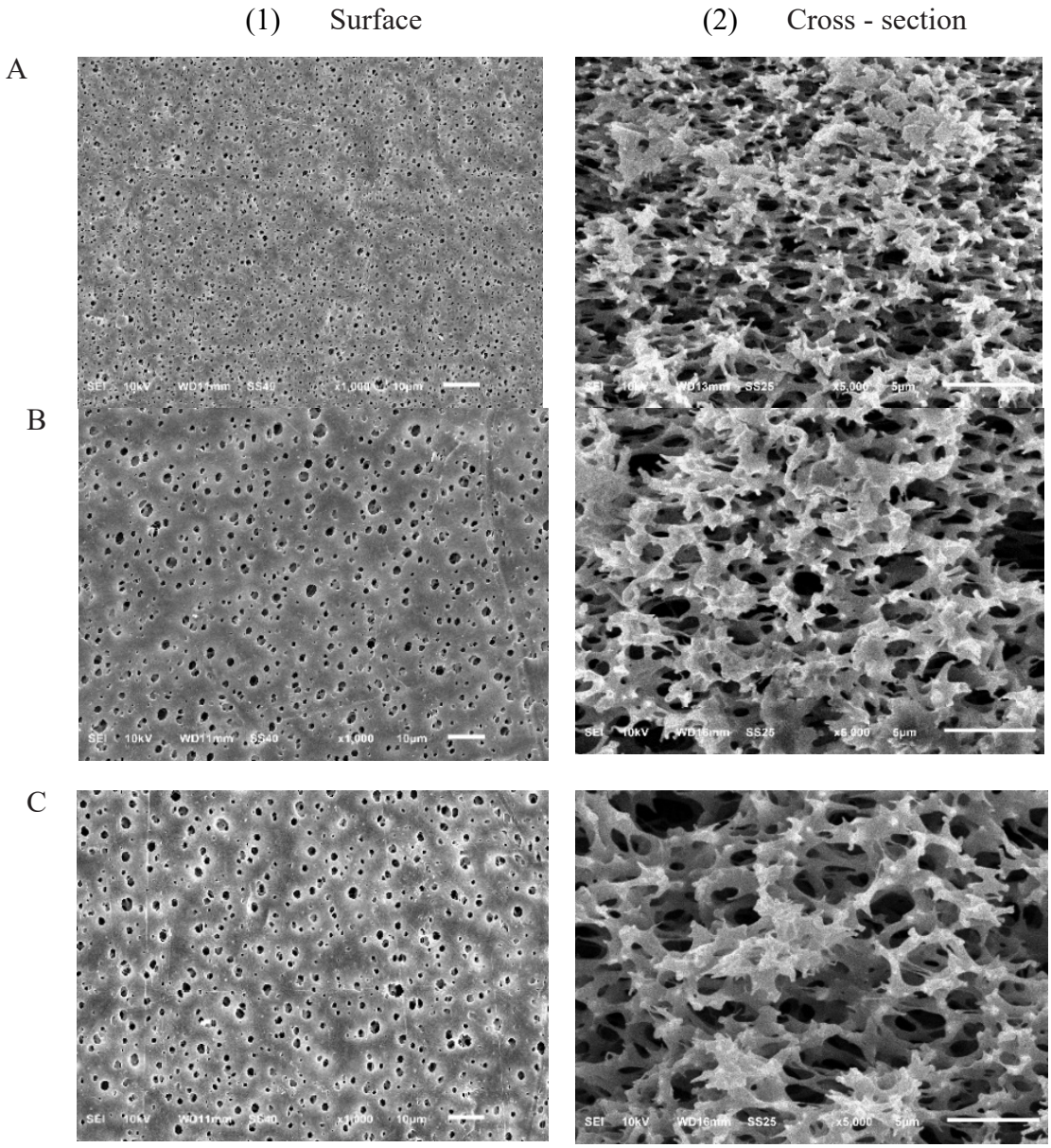


Fig. 1. SEM images of membrane for (A) none, (B) 60 °C; and (C)70 °C of thermal annealing.

pore regularity and interconnectivity are notably improved in thermally treated membranes compared to the none - thermal annealing one.

PES is an amorphous polymer characterized by a glass transition temperature and the absence of a distinct melting point due to the lack of crystalline phases [32]. When heated, the amorphous zones in PES become more flexible and undergo structural relaxation, which enables pore enlargement and improved pore connectivity. Thermal annealing at 60°C and 70°C induces low softening and limited relaxation, which is sufficient to alter the pore structure.

Pore enlargement after thermal annealing in PES can be explained by several mechanisms, including the relaxation of internal stresses within the membrane structure, molecular reorganization that forms more open pore pathways, increased chain mobility in the amorphous regions, and the effect of residual solvent physically expanding the pores [10, 31]. After phase inversion, membranes typically retain internal stress due to rapid phase changes. Thermal annealing provides adequate time and energy for the release of these stresses,

allowing previously compressed or restricted pores to expand [14]. Enhanced polymer chain mobility in the amorphous phase also helps soften the structure and reorganize the pores. Additionally, residual solvent or water trapped within the membrane may expand during heating, contributing to physical pore enlargement [38].

The effect of thermal annealing on functional group of membrane

Fourier Transform Infrared Spectroscopy analysis was conducted to evaluate whether low - temperature thermal annealing caused detectable changes in the chemical structure of the PES membrane. As shown in Fig. 2, the FT - IR spectra of the non - annealed membrane and the membranes annealed at 60°C and 70°C exhibit generally similar absorption patterns. The main characteristic peaks of PES are observed in all samples, including aromatic ring vibrations at approximately 1580 - 1500 cm^{-1}, asymmetric and symmetric stretching vibrations of sulfone groups (- SO_2 -) near 1320 and 1150 cm^{-1}, and C- O - C ether stretching around 1240 cm^{-1} [13, 39, 40]. Similar characteristic absorption bands have also been reported in previous PES - based membrane studies, confirming that these peaks are associated with the intrinsic chemical structure of the PES backbone rather than newly formed functional groups [13, 39, 40].

No new absorption peaks, peak disappearance, or clear spectral shifts were observed after annealing at 60°C or 70°C. This result indicates that the mild thermal treatment applied in this study did not induce detectable chemical degradation or the formation of new functional groups in the PES membrane. This finding is reasonable because the annealing temperatures used in this study were far below the glass transition temperature of PES; therefore, the treatment was not expected to provide sufficient thermal energy to break or form chemical bonds within the polymer backbone [13, 27]. In this respect, the present result differs from studies involving chemical crosslinking, inorganic incorporation, or other chemical modification routes, where FT - IR spectra may show additional bands or more pronounced changes due to the introduction of new functional components into the membrane matrix [25, 27, 39 - 41].

The absence of major spectral changes is also consistent with previous thermal annealing studies reporting that annealing may modify membrane performance mainly through physical rearrangement of polymer chains, changes in free volume, pore structure adjustment, or improved chain packing, rather than through direct alteration of chemical composition [27, 31, 32, 41]. For example, thermal annealing has been reported to influence transport properties and membrane compactness by changing the aggregation structure and free - volume distribution of polymer membranes [31, 32]. Similarly, studies on thermally treated polymeric membranes have shown that changes in permeability or selectivity can occur even when the main chemical structure remains unchanged [27, 41]. Therefore, the similar FT - IR spectra observed in this study support the interpretation that low - temperature annealing acted primarily as a physical post - treatment.

Although minor variations in absorption intensity may be observed in Fig. 2, these differences are not sufficiently distinct to support a detailed interpretation of increased functional group exposure, enhanced hydrophilicity, or stronger molecular ordering. Such small variations may also arise from differences in sample thickness, surface contact during FT - IR measurement, or local membrane heterogeneity. Therefore, unlike the previous version of the discussion, the FT - IR result is not used here as direct evidence of significant chemical modification. Instead, it is interpreted as evidence that the PES membrane maintained its chemical integrity after annealing.

Based on this interpretation, the changes observed

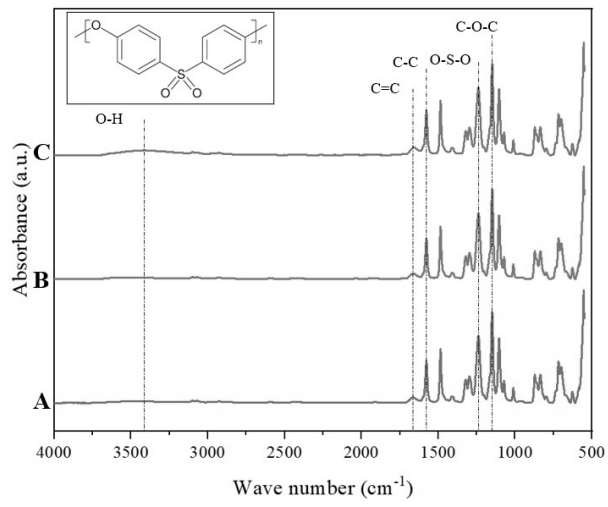


Fig. 2. FT - IR spectra of (A) none, (B) 60°C, and (C) 70°C thermal annealing of PES membrane.

in membrane morphology, mechanical properties, and pure water flux are more appropriately attributed to physical structural rearrangement, including limited polymer chain relaxation, improved pore connectivity, and possible changes in free - volume distribution. This explanation agrees with previous reports showing that thermal annealing can affect membrane structure and transport behaviour without necessarily producing new FT - IR peaks, particularly when the treatment is performed under conditions that do not chemically degrade the polymer matrix [27, 31, 32, 41]. Thus, the FT - IR results in this study confirm the chemical stability of PES after low - temperature annealing, while the performance changes are more strongly associated with physical modification of the membrane structure.

The effect of PES thermal annealing to the mechanical strength

The effect of thermal treatment on tensile strength and elongation at break was analysed to determine the extent to which the thermal annealing process can modify the physical properties of PES membranes. Based on Fig. 3, it is evident that thermal annealing at different temperatures has a significant effect on the tensile strength and elongation at break of PES membranes. Tensile strength increased from membrane A (no thermal annealing) to membrane B (annealed at 60°C) but decreased in membrane C (annealed at 70°C). Conversely, elongation at break progressively declined with increasing annealing temperature. The increase in tensile strength at 60°C reflects improved polymer chain ordering because of low thermal annealing. This finding is consistent with the FT - IR spectra, which show enhanced intensity of aromatic and polar functional group peaks, indicating stronger molecular interactions and structural reorganization. SEM images of membrane B (60°C) also display more uniform and interconnected pore structures, which may contribute to better stress distribution during tensile testing. In membrane C, SEM reveals larger pore sizes compared to membrane A, with a less dense and more open structure, making it more prone to crack initiation. Thermal annealing at this temperature may approach the threshold of excessive structural relaxation, leading to reduced mechanical integrity despite improved water flux [28, 42].

The decrease in elongation at break from membrane A to C indicates that the membrane becomes stiffer and less elastic. This is attributed to increased polymer chain ordering and the loss of flexibility in the amorphous structure, as supported by the intensified FT - IR bands associated with aromatic and sulfone groups. While thermal annealing enhances internal bonding through structural relaxation, it compromises flexibility, thereby reducing the maximum deformation of membrane before failure [38].

Pure water flux performance

Permeate flux is a key parameter in evaluating membrane separation efficiency, as the rate of fluid flow through the membrane greatly influences the capacity and productivity. A high flux value indicates the ability of membrane to transport a large volume of water within a given time, directly impacting operational effectiveness and processing costs. This subsection discusses

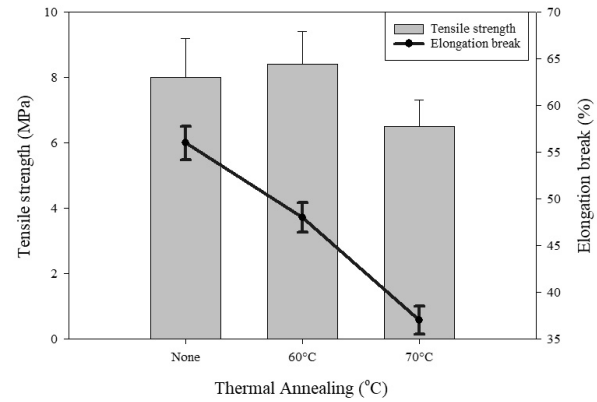


Fig. 3. Tensile strength and elongation break of (A) none, (B) 60°C, and (C) 70°C thermal annealing of PES membrane.

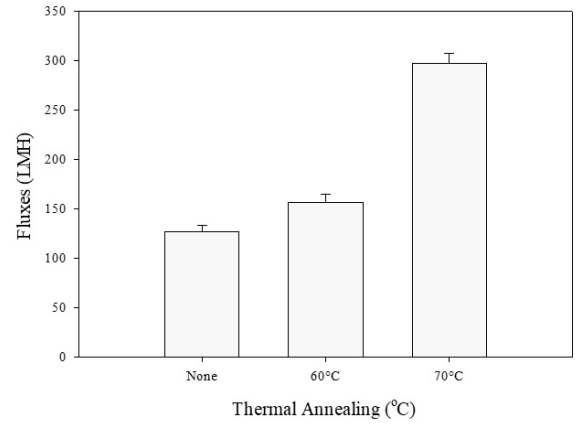


Fig. 4. Pure Water Flux Performance of (A) none, (B) 60°C, and (C) 70°C thermal annealing of PES membrane.

membrane characteristics in terms of flux performance using distilled water and correlates the findings with other data presented in the previous subsections.

Fig. 4 shows that the pure water flux increases with higher thermal annealing temperatures, with Sample C exhibiting the highest flux. This increase is primarily attributed to the enlargement of pore size within the membrane structure, which reduces flow resistance and facilitates easier water passage. However, an excessively open structure may negatively impact filtration efficiency by reducing the selectivity of membrane for contaminants or smaller molecules. This finding aligns with SEM images, which visually demonstrate significant morphological changes due to thermal annealing. At 60°C (A), the membrane exhibited a compact morphology with small, uniformly distributed pores, reflecting a more controlled and closed structure. In contrast, at 70°C (C), pore enlargement and greater pore density were observed, indicating a more open and highly porous structure, directly correlating with increased porosity and water flux.

FT - IR spectra reveal that thermal annealing influences intermolecular interactions within the PES polymer matrix. At 60°C, hydrogen bonding and intermolecular forces remain relatively stable, preserving polymer integrity. However, at 70°C, a decrease in the vibrational band intensities associated with -SO_2- and -C=O groups suggests reduced polymer chain ordering and weakened segmental interactions. This indicates that high - temperature annealing promotes chain relaxation, increasing segmental mobility, enlarging pore size, and enhancing water permeability. Therefore, FT-IR findings reinforce SEM observations, showing that increased annealing temperature results in a looser structure and improved water flux performance.

Mechanical property data indicate an inverse relationship (trade - off) between water flux and mechanical strength. As water flux increases, tensile strength and elongation at break decrease. Membrane A and B, annealed at lower temperatures (60 - 65°C), show high tensile strength (~ 8 - 9 MPa) and good elongation values (> 50 %), indicating strong and flexible membrane structures. In contrast, Membrane C, treated at the higher temperature of 70°C, exhibits a sharp increase in water flux (~ 300 LMH), but a significant decline in tensile strength (~ 6 MPa) and elongation (~ 37 %). This reduction results from increased rigidity due to weakened intermolecular bonding, making the membrane more brittle and mechanically fragile. This emphasizes that improvements in water permeability must be carefully balanced to avoid compromising the structural durability of membrane.

The substantial increase in pure water flux in membrane C demonstrates the effectiveness of thermal annealing in enlarging pores and enhancing permeability. However, this benefit comes at the expense of membrane strength and elasticity. SEM and FT - IR data indicate that a more open structure and weakened chemical interactions support higher flux but must be balanced with mechanical stability. Therefore, excessively high annealing temperatures like 70°C should be avoided for applications requiring long - term mechanical durability. Moderate temperatures, such as 60 - 62°C, offer the best compromise between water flux performance and the structural integrity of PES membranes.

CONCLUSIONS

This study demonstrates that low - temperature thermal annealing influences the characteristics and performance of PES membranes. Thermal annealing at 60°C improved pore structure regularity, morphological compactness, and mechanical strength without detectable changes in the chemical structure of the membrane, as confirmed by SEM, FT - IR, and tensile testing analyses. In contrast, annealing at 70°C resulted in higher pure water flux but reduced tensile strength and elongation at break, indicating a more open and mechanically weaker membrane structure. Therefore, among the annealing temperatures evaluated in this study, treatment at 60°C provided a more favourable balance between mechanical strength and water permeability than treatment at 70°C. However, because only two annealing temperatures were investigated, this condition should not be regarded as the optimal annealing temperature. Further studies using a broader and more closely spaced temperature range are required to determine the true optimum thermal annealing condition for PES membranes.

For further implementation, extended testing using actual wastewater and rejection analysis of complex contaminants such as dyes, heavy metals, and surfactants is necessary to comprehensively evaluate the filtration effectiveness of the membrane. In addition, future studies should examine a wider annealing temperature range

and different annealing durations to better understand the relationship between thermal treatment conditions, membrane structure, mechanical stability, and filtration performance.

A technical and economic feasibility analysis of low - temperature thermal annealing is also essential to assess its applicability at small and medium scale industrial levels. Such analysis would help determine whether this method can be adopted as an efficient, energy saving, and sustainable post - treatment strategy for wastewater treatment applications.

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Authors' contributions

M.M.: conceptualization and methodology; R.A.L.: writing-original draft preparation-review and editing; F.C.: formal analysis; C.D.M.: laboratory work; S.A.P.: laboratory work; N.A.: laboratory work; H.H.: data curation.

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