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SYNTHESIS OF POLYETHERSULFONE/TITANIUM DIOXIDE MEMBRANES: ANALYSIS OF MORPHOLOGY, MECHANICAL PROPERTIES, AND WATER FILTRATION PERFORMANCE

Agung Mataram
Corresponding author
Doctor of Philosophy (PhD), Associate Professor*
E-mail: amataram@unsri.ac.id
Aneka Firdaus
Master of Engineering, Lecturer*
Muhammad Yanis
Doctor of Philosophy (PhD), Associate Professor*
Rahma Dani
Master of Physics Education, Student
Department of Physics Education**
Subriyer Nasir
Doctor of Philosophy (PhD), Professor
Department of Chemical Engineering**
Ahmad Fauzi Ismail
Doctor of Philosophy (PhD), Professor***
Mohd Hafiz Dzarfan Othman
Doctor of Philosophy (PhD), Professor***
*Department of Mechanical Engineering**
**Universitas Sriwijaya
Palembang-Prabumulih str., KM 32, Inderalaya, Indonesia, 30862
***Advanced Membrane Technology Research Centre (AMTEC)
Universiti Teknologi Malaysia
Skudai str., 29a, Johor Bahru, Johor, Malaysia, 81310

The increasing demand for clean water, driven by population growth, urbanization, and industrial activities, has led to significant challenges in public health, the economy, and the environment. Effective water purification technologies are essential to address this issue. This study explores using polyethersulfone (PES) polymer-based membranes reinforced with titanium dioxide (TiO_2) nanoparticles for antifouling applications. The membranes were fabricated using an electric field treatment method. Scanning Electron Microscopy (SEM) revealed a pore size distribution between 1,170 µm and 7,122 µm, demonstrating that this method can be adjusted to create membranes with specific filtration characteristics. Atomic Force Microscope (AFM) analysis showed surface roughness between 150 and 500 nm, indicating that the membrane's surface morphology can be customized to improve performance. Mechanical testing showed that the tensile strength of the membranes varied with the addition of TiO_2: the pure PES membrane (TI0) had a tensile strength of 2.12 MPa, while the TI1 membrane (20 % PES, 1 % TiO_2) exhibited a decrease to 1.84 MPa. The TI2 membrane (30 % PES, 1 % TiO_2) showed an increase in tensile strength to 3.86 MPa, confirming the reinforcing effect of TiO_2 on the membrane's mechanical properties. Clean Water Permeability (CWP) testing indicated flux values of 2558.9 $L/m^2 \cdot h \cdot bar$ for TI0, 1263.1 $L/m^2 \cdot h \cdot bar$ for TI1, and 2763.9 $L/m^2 \cdot h \cdot bar$ for TI2, highlighting the optimal balance of mechanical strength and permeability in TI2. The PES/TiO_2 composite membrane, made using an electric field method, shows promise for water filtration due to its enhanced permeability, providing an efficient solution for water treatment

Keywords: antifouling, fabrication, filtration, membranes, permeability, polyethersulfone, titanium dioxide

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1. Introduction

In recent decades, the demand for clean water has increased significantly due to rapid population growth, urbanization, and industrial activities. According to UNICEF and the World Health Organization (WHO), more than two billion people worldwide do not have access to safe drinking water [1]. Many are forced to use polluted water sources, which often contain contaminants, such as pathogenic bacteria, toxic chemicals, and microscopic particles [2]. This condition not only adversely affects public health but also increases the economic burden and exacerbates environmental degradation [3]. Therefore, efficient, environmentally friendly, and economical water purification technologies are urgently required to overcome this clean water crisis.

One promising approach in water purification is membrane technology, specifically for water separation and filtration applications. Polymer-based water filtration membranes have evolved as effective solutions because of their ability to remove various contaminants while maintaining high water flow. This technology offers several advantages, such as lower energy usage than conventional methods and high separability [4]. Among the various types of membrane materials available, polyethersulfone (PES) is one of the most widely used polymers owing to its stable thermal and chemical properties, ease of processing, and suitability for various separation applications [5]. It is resistant to many types of solvents and exhibits good thermal stability.

However, while PES has a number of advantages that favor its use in water filtration technology, it also has some limitations. One of the main disadvantages of pure PES membranes is their hydrophobic nature [6]. This property makes PES likely to attract organic contaminants and microorganisms that cause fouling, that is, the accumulation of particles or compounds on the membrane surface. Fouling is a common problem in membrane applications, as it can impede water flow, lower filtration efficiency, and shorten the membrane lifetime. This fouling problem not only increases the cleaning frequency and operating costs but also reduces the efficiency of the filtration technology.

To overcome these limitations, researchers have developed various methods to improve the properties of PES membranes, one of which is to add additional materials or nanoparticles to the membrane structure. The addition of nanoparticles to PES membranes is expected to improve their properties such as hydrophilicity, resistance to fouling, and mechanical strength. Among the various types of nanoparticles, titanium dioxide (TiO_2) has attracted considerable attention in membrane research because of its favorable properties. TiO_2 is a photocatalytic material that can generate free radicals when exposed to light, which allows the degradation of organic compounds attached to the membrane surface [7]. In addition, TiO_2 has antimicrobial properties that can reduce the growth of microorganisms on the membrane surface, thereby reducing fouling problems and extending the service life of the membrane. TiO_2 also has hydrophilic properties that can increase water permeability through the membrane, which is very important in water purification applications.

2. Literature review and problem statement

In recent years, many studies have focused on improving the performance of PES/TiO_2 membranes through various modification methods. One of the methods that is often used is phase inversion and electrospinning. Through the phase inversion method, ultrafiltration membranes based on Polyethersulfone (PES) with embedded TiO_2 particles have been successfully synthesized for color filtration [8]. The PES/TiO_2 composite membrane showed improved water permeability and good color filter rejection results of 15.1–34.7 % compared to the pure PES membrane. This method has been proven to increase water permeability but faces challenges related to porosity, which is difficult to control because it involves a liquid-to-solid phase change. Another approach employs electrospinning to produce more structured PES/TiO_2 membranes with high hydrophilicity [9]. As a result, the TiO_2 content in the PES nanofibers was effective against phenol degradation. However, this method often requires complex parameter control and specialized equipment. Another modification technique that has been used for composite membranes is the layer-by-layer (LBL) motto, which offers many advantages in terms of control and layer structure. Surface modification of PES membranes with a GO-TiO_2 layer through this method has been shown to enhance photocatalytic performance [10]. This method offers many advantages, but the process is inefficient because it is time-consuming, and the complexity of its control and vulnerable adhesion of the coating are challenges. Other studies have successfully made polyvinyl Alcohol-based cation exchange membranes using the sol-gel method to improve alkali recovery through diffusion dialysis [11]. The Sol-Gel method does have the advantage of adding nanoparticles of organic matter to the membrane. Still, it requires a high temperature for drying, and it isn't easy to control the pores quickly. Then, another research team made a new nanocomposite membrane with a continuous proton channel and reinforcement network using the Electrospinning Solution Casting combination method [12]. Combining these two methods produces fibers with high mechanical strength, but it has limitations due to the usually complicated process. Other studies have successfully fabricated recycled high-density polyethylene (HDPE) membranes and virgin polyethylene powder combined with calcium carbonate fillers using the Stretch-Induced Spin-Cast method for microfiltration membrane applications [13]. This method has the advantages of controlling the pore structure, having good mechanical properties, and adapting to specific applications. However, its drawbacks include process complexity, specialized equipment needs, and spin speed dependence. Other studies developed polysulfone membranes by vapor-induced phase separation method for oil/steam filtration [14]. However, this method requires a long process time, potential residual solvent effects, and not fully uniform pores. Today, researchers are constantly looking for methods that not only improve membrane performance but can also be produced economically and easily implemented on a large scale. Thus, the Electric Field method offers an attractive approach with low cost and fast processing to improve the quality of PES/TiO_2 membranes. This method allows for a more uniform distribution of TiO_2 nanoparticles in the membrane matrix, potentially improving the filtration performance. In addition, this technique offers advantages in the control of membrane morphology, such as pore distribution and thickness, which can improve membrane permeability and selectivity.

The use of the Electric Field method in the fabrication of PES/TiO_2-based membranes promises significant improvements compared to conventional methods, especially in overcoming the limitations of filtration performance, mechanical stability, and energy efficiency that are not yet optimal in current filtration membranes. Although the integration of TiO_2 nanoparticles in PES matrices has been known to improve morphological, mechanical, and filtration performance properties, a deep understanding of how the Electric Field method can control the distribution of TiO_2 more precisely remains an unresolved gap. This is important because conventional fabrication methods often fail to achieve the optimal combination of controlled morphology, superior mechanical properties, and efficient filtration performance required in real applications. Therefore, an in-depth study is needed on the fabrication of PES/TiO_2 membranes using the Electric Field method, which can provide better control of morphological structures, improve the distribution of TiO_2 nanoparticles, and ultimately produce membranes with more efficient filtration performance, high selectivity, and better durability.

3. The aim and objectives of the study

This study aims to synthesize and evaluate polyether sulfone (PES) membranes enhanced with titanium dioxide (TiO_2) nanoparticles, prepared using the Electric Field method, to optimize their morphology, mechanical properties, and water filtration performance. The findings from this study are expected to provide practical benefits, such as improving the efficiency and effectiveness of water filtration processes.

To achieve this aim, the following objectives are accomplished:

– to investigate the effect of TiO_2 incorporation on the morphological characteristics of PES membranes based on scanning electron microscope;

– to investigate the effect of TiO_2 incorporation on the topographic morphological characteristics of PES membranes based on Atomic Force Microscope;

– to assess the impact of TiO_2 integration on the mechanical properties of PES membranes;

– to evaluate the water filtration performance of PES/TiO_2 membranes, focusing on their permeability and selectivity.

4. Materials and methods

4. 1. Object and hypothesis of the study

The object of the study is the PES membranes reinforced with TiO_2 nanoparticles for water filtration applications, focusing on their morphology, mechanical properties, and permeability.

The main hypothesis is that incorporating TiO_2 nanoparticles into PES membranes, combined with electric field treatment, will enhance the membranes' antifouling properties, mechanical strength, and permeability, making them more effective for water filtration.

The assumptions made include that the electric field treatment method ensures uniform distribution of TiO_2 nanoparticles in the PES matrix, the TiO_2 concentration and fabrication conditions will influence the mechanical and filtration performance, and the membranes' antifouling and permeability performance will remain stable under typical water filtration conditions.

The simplifications adopted in the study include evaluating only three membrane compositions (TI0, TI1, and TI2) with varying PES and TiO_2 content, assuming ideal laboratory conditions without considering real-world variables such as temperature fluctuations or water quality variations, and limiting mechanical testing to tensile strength without assessing other types of mechanical stress like shear or compression.

4. 2. Materials

PT. Dira Sonita (Palembang, Indonesia) supplied the Polyethersulfone (PES), Dimethylformamide (DMF, CAS 68122, anhydrous, 99.8 %), and Titanium Dioxide (TiO_2) additives, which were utilized as received without additional processing.

4. 3. Membrane preparation

The membranes were prepared in three specimens with different weight ratio fractions (wt.%) and different polymer blends for each sample, that is, PES 20 %, PES 25 %, and PES 30 %. The use of a high polymer concentration can strengthen the structure and pore bonding of the membrane, which will have a positive impact on the mechanical and morphological properties of the membrane. The addition of TiO_2 was the same in each sample, that is, 1 % in 20 % and 30 % PES. In our previous research, the addition of TiO_2 at a concentration of 1 % was the optimal membrane-reinforcing agent [15]. PES with a 25 % concentration without TiO_2 mixture was used as a comparison specimen. The schematic of the PES/TiO_2 membrane experiment can be seen in Fig. 1. Schematic of the PES/TiO_2 membrane experiment.

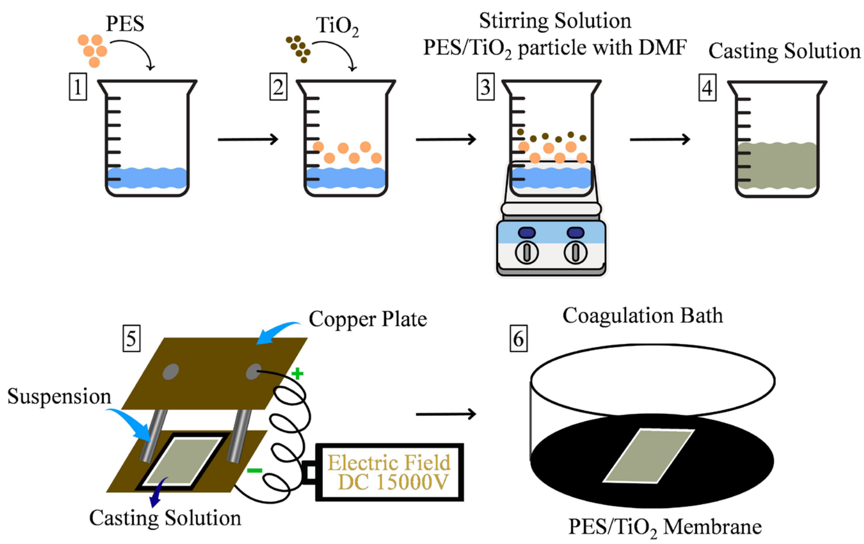

Fig. 1. Schematic of the PES/TiO_2 membrane experiment

The initial process involved dissolving PES and DMF and then mixing TiO_2. Mixing using a magnetic stirrer, at a temperature of approximately 40 °C for 8 h until homogeneous, PES is poured into an airtight glass that aims for the deposition process, and review of the solution is not mixed. The electric Field was prepared as the latest innovation in membrane manufacturing. The research was conducted with a copper plate that was electrified by a 15 kV DC current electric field, and a plaster was used for mold boundaries. The process of making specimens is a common method used in membrane manufacturing, such as inversion phase molding, electric field current flowing, and immersion in water. The specimen was flat. The mixture that has passed the deposition stage continues the inversion-phase molding process. Then, a 15 kV DC current was applied for 2 min. Furthermore, immersion was performed until the specimen was detached from the copper plate and dried at room temperature for 24 h. The membrane mixing composition can be seen in Table 1.

Table 1

The composition of PES/TiO_2 precursor solution

Sample	PES (g)	TiO_2 (g)	DMF (g)
TI0	12.5	0	37.5
TI1	10	0,1	39.9
TI2	15	0,15	34.85

Polyether sulfone (PES) was chosen as the primary material due to its high-temperature resistance, good chemical stability, and high compatibility in membrane applications. In sample T0, PES was used additionally without TiO_2 to produce a membrane control that could be used as a reference in propagating the effect of TiO_2 addition. Samples T1 and T2 showed a gradual increase in TiO_2 concentration, 0.1 g, and 0.15 g, respectively, which is expected to improve the functional properties of the membrane, such as mechanical strength, hydrophobic properties, and antimicrobial ability. In addition, the different DMF solvent ratios in each sample were designed to maintain the homogeneity of the solution, which is very important in the membrane manufacturing process through the phase inversion method. The combination of PES, TiO_2, and DMF in various proportions aims to affect the membrane's characteristics.

4. 4. Characterization

The surface morphology and structure of the membrane were observed using an SEM (Inspect S50, FEI Company) operated at a voltage of 15.00 kV. The surface of the sample was coated with gold prior to SEM analysis. Hardness and roughness testing of the membrane was carried out using Atomic Force Microscopy (AFM AMTEC, UTM) in a three-dimensional form with a scanning area of 10×10 µm. The mechanical properties were determined using a tensile tester (ZWICK ROEL Material Testing Machine Type BT2-FR020TH. A60, Germany) according to the ASTM D638.05/2008 testing standards. The performance of the membrane water filter was determined by normalized clean water permeability (CWP, Indonesia) through a cassette cleanliness test. This involves measuring the flow of clean water through a membrane under standard pressure and temperature conditions. The membrane was wetted in ultrapure water for 15 min, pumped to a pressure of 1 bar, and its performance was measured using a 50 mL measuring cup for 1 h.

5. Research results on morphology, mechanical properties, and water filtration of polyether sulfone/titanium dioxide membrane

5. 1. Morphology of polyether sulfone/titanium dioxide membrane based on scanning electron microscope results

Morphological analysis of the membrane showed that the TI0 membrane had a varied pore distribution with diameters between 2.042 to 7.122 µm, reflecting a heterogeneous macropore structure. The results of the scanning electron microscope of the membrane can be seen in Fig. 2.

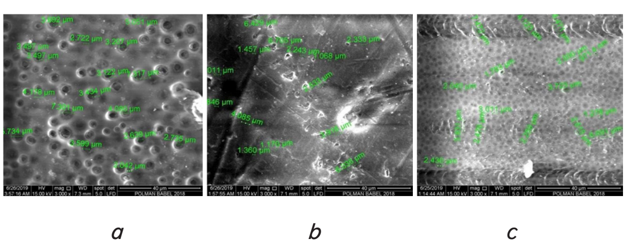

Fig. 2. Scanning electron microscope of membrane PES/TiO_2: *a* – TI0; *b* – TI1; *c* – TI2

The addition of TiO_2 to the TI1 membrane led to a reduction in pore size to 1.170 to 6.705 µm, with agglomerations of TiO_2 particles dispersed on the membrane surface. The interaction between the TiO_2 particles and the PES matrix resulted in a denser structure with smaller pores. In the TI2 membrane, with a higher concentration of PES polymer (30 %) and 1 % TiO_2, the membrane structure became more compact with a pore size of 2.438 to 4.133 µm. This increased the mechanical strength and selectivity of the membrane, although it reduced the water permeability. In addition, modification of the membrane surface using the Electric Field method at a voltage of 15 kV resulted in a smoother membrane surface, reduced pore size, and lowered the degree of concentration polarization, thereby improving flow and reducing fouling.

5. 2. Morphology of polyether sulfone/titanium dioxide membrane based on atomic force microscopy results

The membranes' surface morphology and topographic characteristics were evaluated using Atomic Force Microscopy to see the impact of TiO_2 addition on the membrane structure. This technique provides a detailed picture of the surface roughness, particle distribution, and level of topographic homogeneity of each membrane. This analysis is critical to understanding how the addition of TiO_2 affects the physical properties of the membrane, including its permeability, selectivity, and mechanical stability. The results of the atomic force microscopy of the membrane can be seen in Fig. 3.

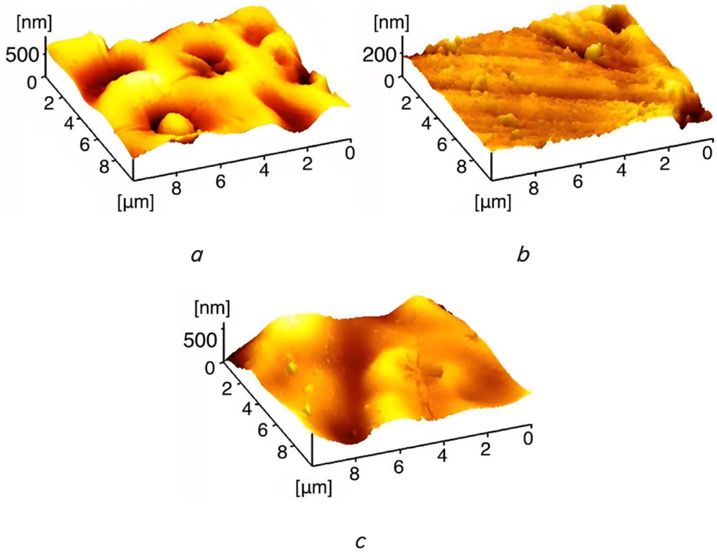

Fig. 3. Atomic force microscopy images of PES/TiO_2 membranes: *a* – TI0; *b* – TI1; *c* – TI2

Analysis using atomic force microscopy shows that the TI0 membrane has a surface with deep, sharp pores and a surface roughness of 500 nm. This structure allows high water filtration capacity but with limited selectivity. The TI1 membrane showed improved roughness compared to TI0, with maximum roughness reaching 200 nm, but the uneven surface indicated an inhomogeneous distribution of TiO_2 particles, resulting in agglomeration and high variation in topography. In contrast, the TI2 membrane has a smoother surface with a maximum roughness of 150 nm and a more even distribution of TiO_2 particles. This structure results in more defined pores, a more consistent surface, and better interaction between PES and TiO_2, providing a balance between water permeability and mechanical stability.

5. 3. Mechanical properties test results of polyether sulfone/titanium dioxide membrane

Tensile strength testing was carried out to involve the effect of variations in the composition of PES and TiO_2 on the membrane mechanism properties. This method aims to understand how changes in material concentration affect the ability of the membrane to withstand tensile loads. This mechanical strength analysis is important to determine the best composition combination that provides optimal structural stability to the membrane. The results of the mechanical properties of the membrane can be seen in Fig. 4.

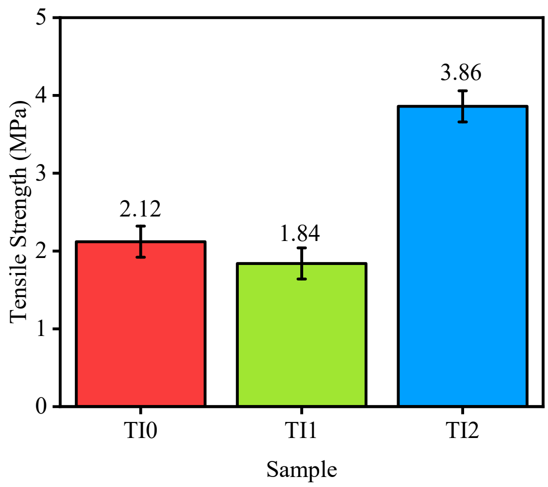

Fig. 4. Mechanical properties of membrane TI0, TI1, and TI2

The TI0 membrane, consisting of pure PES, had a tensile strength of 2.12 MPa, showing moderate mechanical properties as PES is known to have good mechanical resistance but without additional reinforcement. The addition of TiO_2 to the TI1 membrane with 20 % PES and 1% TiO_2 decreased the tensile strength to 1.84 MPa. This decrease is due to the reduction in PES content, which reduces the role of the matrix polymer in supporting mechanical strength. In contrast, the TI2 membrane with PES concentration increased to 30 % and TiO_2 remained at 1 % showed a significant increase in tensile strength, reaching 3.86 MPa. The combination of increasing PES concentration and adding TiO_2 works synergistically to strengthen the membrane, with TiO_2 acting as a reinforcement that improves internal load distribution and creates a stronger bonding network within the polymer matrix.

5. 4. Cleans water permeability test results of polyethersulfone/titanium dioxide membrane

Clean water flux measurements were performed to power the permeability of membranes with various material compositions. This method is essential to understand how variations in PES and TiO_2 concentrations affect the membrane's ability to filter air efficiently. Flux analysis provides information regarding the relationship between the membrane's morphological structure, pore distribution, and permeability performance. The membrane flux graph can be seen in Fig. 5.

Clean water flux measurements showed that the TI0 membrane had a flux of 2558.9 L/m^2.h.bar. This value is quite high due to the porosity formed in the membrane structure during the manufacturing process, although the pure composition of PES is hydrophobic. The TI1 membrane, with a composition of 20 % PES and 1 % TiO_2, showed a significant decrease in flux to 1263.1 L/m^2.h.bar. This decrease was caused by changes in morphological structure and uneven pore distribution, as well as TiO_2 agglomeration that obstructed water flow. In contrast, the TI2 membrane, with a composition of 30 % PES and 1 % TiO_2, showed the highest flux of 2763.9 L/m^2.h.bar. This increase is due to the synergistic combination of a higher concentration of PES, which strengthens the membrane structure, and the addition of TiO_2 which increases surface hydrophobicity and prevents fouling. This combination results in a membrane with a stable pore structure and a more even distribution of TiO_2 particles, providing optimal water permeability and good mechanical stability.

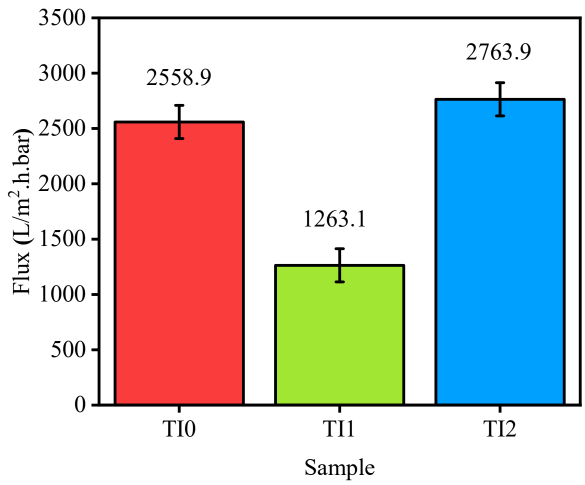

Fig. 5. Graph of PES/TiO_2 membrane flux value

6. Discussion of the research results on morphology, mechanical properties, and water filtration of polyether sulfone/titanium dioxide membrane

Morphological analysis of the membrane was performed using Scanning Electron Microscopy to observe the effects of varying the polymer concentration and TiO_2 addition on the pore structure and membrane surface. Fig. 2, *a* shows that the TI0 membrane has a fairly varied pore distribution with diameters between 2.042 and 7.122 µm. The pores appear to be evenly distributed across the membrane surface, reflecting a macroporous structure. These structures tend to be heterogeneous with relatively large pore sizes, potentially providing high permeability, but with limited selectivity. The large pore size in the membrane with pure PES is due to the low polymer concentration and absence of reinforcement. Under these conditions, the electric field applied during the membrane manufacturing process causes the membrane material to stretch more easily [16]. This is because of the lack of internal resistance in the polymer structure, which allows the material to undergo more significant deformation. As a result, the strain that occurs due to the electric field effect tends to form more evenly distributed pores in the membrane because the polymer structure is not reinforced enough to withstand the external force applied [17].

Fig. 2, *b* shows that the TI1 membrane has a significant reduction in pore size, ranging from 1,170 µm to 6,705 µm, and visible agglomeration of TiO_2 particles scattered on the membrane surface. Several areas of aggregation can be associated with the TiO_2 particles and interact with the PES matrix. The addition of TiO_2 often causes agglomeration because of the natural properties of the particles, which tend to attract each other and agglomerate [18]. This is due to the strong van der Waals forces between the nanometer-sized TiO_2 particles, which tend to interact and form agglomerations [18]. In addition, the distribution of TiO_2 in the polymer matrix can also be affected by the compatibility between the nanoparticles and the polymer matrix. If TiO_2 is not well dispersed in the polymer, it encourages the particles to gather and form agglomerations, which ultimately results in an uneven membrane surface. Meanwhile, the decrease in pores in the membrane is caused by changes in the formation phase and microstructure of the membrane owing to the presence of particles. TiO_2 interacts with polymer chains, which increases the viscosity of the polymer solution [19]. This increase in viscosity slows the process of solvent evaporation and solid phase formation during the membrane molding process, thus creating a denser structure and smaller pores.

The PES membrane with a higher polymer concentration (30 %) and the addition of 1 % TiO_2 in Fig. 2, *c* shows a more compact structure with pore sizes ranging from 2.438 μm to 4.133 μm. This more compact structure is likely to provide increased mechanical strength but potentially reduce water permeability. The compact structure of membranes with higher concentrations and the addition of TiO_2 is thought to be because TiO_2 acts as a nucleation center that encourages the formation of a more compact and uniform matrix [20]. TiO_2 particles dispersed in the polymer solution can fill the gaps between the polymer chains, which in turn reduces the space between the polymer chains required to form more uniform pores [21]. Consequently, the final membrane structure was tighter, with smaller pores and a more uniform pore size distribution. In addition, the compact and evenly distributed pores and the lack of agglomeration in the TI2 membrane are due to the optimal PES concentration of 30 %, in accordance with our previous study [22]. Thus, the membrane formed is also optimal, even with the same addition of 1 % TiO_2, compared to the TI1 membrane. The addition of TiO_2 not only strengthened the membrane structure but also inhibited the growth of large pores, which increased the selectivity of the membrane.

The membrane surface was modified using the Electric Field method at a current of 15 kV. The Electric Field method (jumping sparks) in the membrane manufacturing system has the advantage of reducing the roughness and shrinking the pores of the membrane surface, which aims to reduce the level of concentration polarization and prevent the deposition of pollutants on the membrane surface to reduce fouling and increase the flux on the membrane [23]. In the study for mixed-polymer PES and graphene oxide (GO) with a DC current of 5 kV at the time of membrane formation. This Electric Field method was used so that Silver Nitrate ($AgNO_3$) could spread evenly across the surface of the membrane so that agglomeration did not occur [24]. Agglomeration of TiO_2 nanoparticles during fiber formation is another reason for the ratio formation [25]. However, this process can also reduce permeability, depending on the application needs and the specific formulation of the membrane. Overall, variations in polymer concentration and TiO_2 addition had a significant effect on the morphology and potential performance of the membrane in filtration applications, suggesting that these modifications can be customized to achieve specific filtration characteristics according to application needs.

Atomic Force Microscopy analysis of the PES/TiO_2 membranes under various conditions provides important information on the morphological evolution that affects the mechanical properties and water filtration performance. The morphologies of the TI0, TI1, and TI2 membranes showed significant changes in surface roughness and pore distribution, which had a direct impact on the membrane's ability to facilitate water flow while maintaining optimal selectivity, can be seen in Fig. 3. The TI0 membrane exhibited a significant morphology with deep and sharp pores. The surface roughness was 500 nm. The presence of larger pores allows for increased water filtration capacity by allowing higher flow, yet limited selectivity [26]. This result is consistent with the SEM results, which show large pores. This increase in surface roughness may degrade the mechanical properties, as a very rough surface may introduce structural weaknesses. Nonetheless, the TI0 membrane morphology is optimal for applications that require a high filtration performance, which favors permeability.

In the TI1 membrane, despite the improvement in surface roughness compared to TI0, AFM showed a relatively rough surface with significant height variations, indicating uneven topography. The maximum height reached approximately 200 nm, indicating high surface roughness. This morphology likely limits the filtration efficiency of the membrane because of its uneven surface. The uneven surface and roughness of the TI1 membrane were due to the uneven distribution of TiO_2 in the inhomogeneous PES matrix [27]. Unevenly distributed particles can lead to the formation of agglomerates or lumps, resulting in surfaces with high variations in roughness. This agglomeration resulted in peaks and valleys on the membrane surface, thereby affecting the overall morphology.

In contrast, the TI2 membrane exhibited a smoother surface, with an overall reduction in roughness. The maximum height of the surface decreased to approximately 150 nm, and more defined pores started to form across the membrane. In TI2, the TiO_2 particles are more evenly distributed than in TI0 and TI1, reducing the occurrence of particle agglomeration or clumping [28]. This homogeneity resulted in a more uniform surface with reduced roughness. The good distribution of nanoparticles, such as TiO_2, also helps in the formation of a more regular surface structure, with well-defined pores and a more consistent size. Another factor is the optimization of the solid-to-liquid phase change [29]. In TI2, this process is likely adapted to avoid the formation of rough surface structures. This process also plays a role in creating better interactions between PES and TiO_2, resulting in stronger morphological stability and reduced surface roughness. This improved topography indicates a balance between the surface roughness and pore distribution, which can improve water permeability while maintaining mechanical stability. A smoother surface can also reduce the likelihood of fouling, which is an important factor for long-term filtration applications. Overall, the AFM analysis highlights the important role of surface morphology in tailoring the mechanical properties and filtration efficiency of PES/TiO_2 membranes, with TI2 offering the optimal balance for practical applications.

Fig. 4 shows the tensile strength data for the three membrane types. The TI0 membrane is a pure PES membrane that serves as a baseline for understanding the changes in the mechanical properties when TiO_2 nanoparticles are added. PES membranes are known to have good mechanical properties; however, the increase in their tensile strength is usually limited by the constraints of their chemical structures. The TI0 membrane exhibited a tensile strength of 2.12 MPa. This value can be considered moderate given the nature of PES polymers, which have good mechanical resistance but tend to lack additional reinforcement of filler materials such as nanoparticles. PES is a thermoplastic polymer with advantages in terms of resistance to high temperatures and chemical stability [30]. However, in terms of the mechanical strength, PES is intrinsically influenced by its molecular structure.

The addition of TiO_2 as a reinforcing agent to TI1 and TI2 membranes resulted in two interesting trends. In TI1, where PES was reduced to 20 % with the addition of 1 % TiO_2, the tensile strength decreased to 1.84 MPa. This decrease was due to a significant reduction in the PES content, which reduced the amount of matrix polymer responsible for the mechanical strength. Although TiO_2 is a good reinforcing agent, its presence at low concentrations is not sufficient to compensate for the reduction in PES in terms of tensile strength, which indicates a dependency effect on the base polymer composition [31]. In TI2, the PES concentration was increased to 30 % while maintaining a TiO_2 concentration of 1 %, and a significant increase in the tensile strength was observed, reaching 3.86 MPa. This increase confirms that the combination of increasing PES concentration and adding TiO_2 works synergistically to strengthen the membrane. TiO_2 nanoparticles can act as filler agents that strengthen the composite structure by improving the internal load distribution during strain [32]. In addition, the interaction between PES and TiO_2 at the nanometer scale enables the creation of a stronger bonding network within the polymer matrix, resulting in an increase in the resistance to tensile forces.

Several possible mechanisms are involved in the increase in mechanical strength of TI2. One of the main mechanisms is the formation of physical and possibly chemical bonds between the PES polymer chains and TiO_2 nanoparticles, which increases the cohesion between molecules in the membrane [33]. In addition, TiO_2 nanoparticles can function as stress-dispersing agents, distribute pressure evenly across the membrane, and prevent the formation of microcracks during deformation [34]. This is important because even pressure distribution reduces the risk of premature membrane failure. Overall, the significant increase in the tensile strength of TI2 reflects the optimal combination of PES and TiO_2. These results not only show the importance of adjusting the membrane composition to achieve better mechanical properties but also highlight the important role of TiO_2 as a reinforcing agent in improving the mechanical performance of the membrane. TI2 membranes with superior mechanical properties can contribute significantly to the development of membrane applications that require high tensile strength, resistance to mechanical stress, and long-term stability.

The flux data in the graph show the clean water permeability values for the three membrane types. Fig. 5 shows that the TI0 membrane has a flux of 2558.9 L/m^2, h.bar, the TI1 membrane had a lower flux of 1263.1 L/m^2, h bar, while the TI2 membrane shows the highest flux of 2763.9 L/m^2, h.bar. The TI0 membrane shows that although it is composed of 25 % pure PES, the relatively high flux value of 2558.9 L/m^2, h.bar may be due to the porosity generated in the membrane structure during the manufacturing process. PES is a polymeric material that is naturally hydrophobic [30], however, in the membrane structure produced by the electric field technique, pores are formed that allow water to pass through the membrane more easily. However, this permeability can be limited by hydrophobic interactions, which may decrease water absorption efficiency in the long run.

In the TI1 membrane, in which 20 % PES and 1 % TiO_2 were added, the water flux decreased significantly to 1263.1 L/m^2, h.bar. This decrease could be explained by two factors. First, the reduction in PES concentration from 25 % to 20 % may cause changes in the morphological structure of the membrane [35], where the distribution and size of the pores can become more irregular, reducing the water permeability, as described in the previous SEM and AFM analyses. Second, although TiO_2 is known as a hydrophilicity-enhancing agent in membranes [36], at low concentrations and combined with decreased PES, these nanoparticles are not sufficient to significantly increase the permeability. This could be due to unevenly distributed TiO_2 agglomeration, which blocks water flow through the membrane.

In contrast, for the TI2 membrane, which consisted of 30 % PES and 1% TiO_2, the water flux increased to 2763.9 L/m^2, h.bar, which was the highest value among the three samples. This significant increase can be explained by several synergistic factors. First, increasing the PES concentration to 30 % provided better structural strength, which allowed the membrane to maintain a more stable and uniform pore structure. Second, the addition of 1 % TiO_2 to this membrane appears to be effective in increasing the surface hydrophobicity and preventing fouling [37], so that water can pass through the membrane more efficiently. TiO_2 acts as a hydrophilic agent that enhances water absorption [38], and a more uniform distribution of nanoparticles on a stronger PES matrix might improve the membrane morphology [39], optimize the pore size and distribution, and add TiO_2 nanoparticles to determine the permeability properties of the membrane [40]. At TI0, although pure PES resulted in good porosity for water permeability, mechanical performance improvement and pore structure adjustment may be limited without additional reinforcement. The significant decrease at TI1 indicates that, although the addition of TiO_2 could improve hydrophilicity, an excessive reduction in PES concentration may result in a suboptimal membrane structure, causing a drastic decrease in water flux values.

In TI2, increasing the PES concentration to 30 % seems to serve as the main component that maintains the stability of the membrane morphology, whereas the addition of TiO_2 serves as a pore size control agent and enhances the hydrophilicity properties. Combining these two materials at optimal concentrations resulted in a membrane with balanced mechanical and permeability properties, allowing higher water flow through the membrane without compromising the strength of the structure. TiO_2 also helps to reduce the potential for fouling [36] and maintains high water permeability for a longer period of time.

Overall, the optimal combination of PES and TiO_2 on TI2 resulted in a significant increase in clean water permeability, making it an excellent candidate for water filtration applications where permeability efficiency and long-term stability are critical. These results suggest that adjusting the base polymer concentration and adding nanoparticles can be used as strategies to improve membrane performance, particularly in applications that require a balance between mechanical strength and high permeability.

This study has several weaknesses that can be evaluated for future development. One of the main drawbacks is the uneven distribution of TiO_2, which causes agglomeration on the membrane surface, as seen in TI1. This agglomeration hurts the morphology and efficiency of the filtration membrane. To overcome this problem, it is possible to develop dispersion methods for TiO_2 particles, such as high-intensity ultrasonication or surfactants, to ensure a more homogeneous distribution. In addition, modification of TiO_2 through surface functionalization could be a solution to reduce agglomeration tendencies. Another drawback is the influence of PES concentration on membrane porosity. Low PES concentration in TI1 decreases mechanical strength while increasing PES in TI2 improves mechanical properties but potentially reduces permeability if not optimized. Future research can explore the variation of PES concentration with smaller intervals to find a balance point between mechanical strength and permeability. In addition, although the electric field method proved effective in affecting the pore structure and surface roughness, the exact mechanism has not been fully elucidated. Further research on the influence of electric field parameters, such as field intensity and duration, can provide a more in-depth understanding and improve the efficiency of the membrane manufacturing process. With these weaknesses overcome, membranes with optimal performance for water filtration applications can be developed.

Developing research related to PES/TiO_2 membranes can produce innovative solutions in water filtration applications, especially for needs that prioritize efficiency, selectivity, and long-term stability. Further research can focus on optimizing the composite formulation, for example, exploring additional materials such as other nanoparticles (e. g., graphene oxide or carbon nanotubes) to improve the mechanical and hydrophobic properties of the membrane. However, several challenges may hinder the progress of this research. First, the main difficulty is ensuring a homogeneous distribution of nanoparticles within the polymer matrix, as agglomeration of nanoparticles such as TiO_2 can decrease membrane efficiency and result in inconsistent surface properties. Second, the limited laboratory scale may not fully represent the performance of the membrane under actual application conditions, so pilot-scale tests are needed to ensure the validity of the research results. Third, the cost of producing membranes with optimal formulations can be challenging, especially when balancing performance with economics. In addition, the membrane's resistance to long-term fouling, especially in applications involving wastewater or water with high organic content, requires in-depth evaluation to ensure its sustainability.

Using the electric field method, this study successfully synthesized and evaluated polyethersulfone (PES) membranes enhanced with titanium dioxide (TiO_2) nanoparticles.

7. Conclusions

1. The addition of TiO_2 significantly affected the PES membrane's pore distribution and surface structure. The membrane without TiO_2 (TI0) had heterogeneous pores with sizes ranging from 2.042 to 7.122 µm, while the addition of TiO_2 (TI1 and TI2) resulted in smaller pores and a more uniform distribution, with sizes up to 1.170 µm in TI1 and 2.438–4.133 µm in TI2. These results indicate that TiO_2 acts as a pore-controlling agent, improves the membrane structure, and enhances morphological stability through interactions with the polymer matrix.

2. The AFM results showed significant changes in the membrane's surface roughness and topographic distribution. The maximum roughness of the TI0 membrane reached 500 nm, decreasing to 200 nm in TI1 and 150 nm in TI2. The decrease in roughness in TI2 indicated a more even distribution of TiO_2, reduced agglomeration and created a smoother and more uniform surface. This not only improved mechanical stability but also reduced the possibility of fouling, making it optimal for long-term water filtration applications.

3. TI0 had a tensile strength of 2.12 MPa, which decreased to 1.84 MPa in TI1 due to the reduction in PES concentration. However, the tensile strength increased significantly in TI2 to 3.86 MPa, indicating a synergistic interaction between PES (30 %) and TiO_2 (1 %). TiO_2 nanoparticles strengthened the membrane structure through increased cohesion and even stress distribution, making it more deformation-resistant.

4. The membrane permeability showed a significant increase in TI2. TI0 showed a water flux of 2558.9 L/m^2 h, decreasing in TI1 to 1263.1 L/m^2 h due to the agglomeration of TiO_2 that hindered the water flow. In TI2, the flux increased to 2763.9 L/m^2 h. The more stable pore structure, optimal distribution of TiO_2, and increased surface hydrophobicity balance high permeability and good selectivity.

The results showed that the optimal combination of PES concentration and TiO_2 addition could improve the membrane's morphological, mechanical, and filtration performance properties. The TI2 membrane, with its denser structure and uniform pores, has great potential for water filtration applications that require high efficiency and long-term stability.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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Data availability

Data will be made available on reasonable request.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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