# Polysulphone (PSf)-Titanium Dioxide ($TiO_2$) Membranes for Iron (Fe) Removal

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**Abstract.** Laboratory wastewater in PT. Indo Sea Food is a type of waste generated from various laboratory activities consisting of the results or residue of chemical reactions, effects of washing equipment, and consumables from actions that have taken place. The use of chemicals during the activities carried out in the laboratory produces waste containing heavy metals, including the compounds FeCl3 and FeSO4. Oxidized Fe heavy metal waste usually has a red-orange color. It will be hazardous if the waste is directly discharged into the environment and disrupts the ecosystem because of its corrosive nature. Membrane technology is one of the current laboratory waste treatment methods that are efficient, cheap, economical, and accessible. The incorporation of TiO2 into the PSf membrane is expected to increase its hydrophilicity and macro-cavity structure. The purpose of this study was to find out how to make PSF membranes and PSF-TiO2 membranes, to know the characteristics of the membranes and to evaluate the performance of PSF-TiO2 membranes for the separation of Fe2+ metal from PT. Indo Sea Food Rembang. The PSf-TiO2 membrane synthesis begins by preparing a dope solution consisting of PSf as the primary polymer with a composition of 20%. The dope solution was then mixed with 80% N-methyl-2-pyrrolidone (NMP). The membrane was printed using the phase inversion method. The addition of TiO2 was carried out with three different compositions, namely 0.5wt%, 1wt% and 1.5wt%, which aims to determine the optimum condition of the membrane from various variations. The effect of the addition of TiO2 on the performance of the membrane can increase the flux and % of membrane rejection in the composition of the expansion of 0.5wt% TiO2 (PSF-TiO2 0.5wt%).

**Keywords:** Membrane · Polysulfone · $TiO_2$ · waste · $Fe^{2+}$

## 1 Introduction

The use of chemicals during the activities carried out in the laboratory produces waste containing heavy metals, including the compounds FeCl3 and FeSO4. Fe ions are dissolved in the form of Ferro (Fe2+), which is easily oxidized to iron in the form of Ferry (Fe3+) due to exposure to oxygen in the air (Liang et al., 2019). Waste containing Fe ions exceeding its threshold can be very harmful to health and the environment because it causes turbidity in the water. The primary material for the manufacture of membranes is a polymer. Polysulfone is a polymer widely used as the primary material for making


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membranes. Titanium dioxide (TiO2) has been widely used in the study of nanoparticle materials for membrane modification due to its unique properties, such as strong hydrophilicity, chemical stability, commercial availability, and photocatalytic behavior (Sotto et al., 2011). The utilization of TiO2 as a PSf membrane additive demonstrates high hydrophilicity, permeability, and anti-fouling performance in laboratory wastewater filtration. In this study, a study will be carried out with Polysulphone (PSf)-Titanium Dioxide (TiO2) membrane for Iron (Fe) removal. The PSf-TiO2 membrane will be evaluated in detail on its characteristics, including permeate flux, pollutant rejection, and anti-fouling behavior. Membrane stability in terms of flux recovery after membrane cleaning was evaluated in this study. Laboratory wastewater treatment of PT. Indo Sea Food aims to make the waste eligible to be disposed of in the environment.

Wastewater is produced by industrial processes that are liquid and contain suspended solid or dissolved solids that will undergo a process of physical, chemical and biological change. This wastewater produced toxic substances also can disturb or risk of disease and environmental damage.

### 1.1 Food Industry Wastewater

Several factors can influence the characteristics of food industry wastewater. These factors include the source and type of pollutants in wastewater. Generally, wastewater from the food industry has a very complex composition, consisting of compounds and suspense from organic and inorganic materials. The content of organic matter in wastewater from the food industry is primarily fat/oil in a relatively large amount.

### 1.2 Impact of Ferrous Metal Contaminants (Fe) on Health

Ferrous metal (Fe) is an essential heavy metal whose presence in specific quantities is needed by living organisms. However, if heavy metals enter the body excessively, they will turn their function into poison for the body. Iron is a component of hemoglobin in which about 75% of its presence allows red blood cells to carry oxygen and deliver it to body tissues.

### 1.3 Wastewater Treatment Methods Containing Iron Metal (Fe)

So far, the use of wastewater treatment containing ferrous metals has been carried out through conventional techniques such as adsorption, ion exchange, and electrochemical reduction (Asghar et al., 2012). In addition to some of these methods, recently, the use of membrane technology is also being studied because it has the quality to treat wastewater more optimally (Wei et al., 2021).

## 2 Methodology

The tools used are stirring rods, scale analyzers, desiccators, beker glass, Erlenmeyer 50 mL, spatula, volumetric flask, magnetic stirrer, filter paper, centrifuge, and hot plate stirrer. Meanwhile, equipment for membrane characterization will later be used scanning electron microscope (SEM) JSM-6510LA and Fourier Transform Infra-Red (FTIR) Shimadzu prestige 21, and Atomic Absorption Spectrophotometer (SAA) AMT3802AA.

### 2.1 Material

The ingredients used in this study include polysulfone (Merck) as a membrane material, Nmethyl-2-pyrrolidone (NMP) (Merck) as a polymer solvent, TiO2 (Merck) as an additive (0.5% wt, 1% wt, and 1.5% wt), deionized water for sample solvents, and PT laboratory waste samples. Indo Sea Food with a concentration of Fe2+ ions of 250 ppm.

### 2.2 Research Methodology

This study synthesized PSf and PSf-TiO2 membranes and compared their characteristics and ability to treat wastewater containing ferrous metals. The features of the PSf and PSf-TiO2 membranes were analyzed for morphological properties and states using SEM, analyzed for functional group content and interactions using FTIR, and analyzed for their properties to water through contact angle analysis. In addition, the performance of the PSf and PSf-TiO2 membranes also needs to be examined by calculating the influx determined by - filtration cells. Furthermore, the membrane will be tested for its ability to treat wastewater containing the heavy metal Fe. Samples were taken from PT. Indo Sea Food, Rembang.

## 3 Results and Discussion

Synthesis of PSf-TiO2 membranes with various TiO2 variation ranges. The synthesized membrane is then tested for contact angles to determine the characteristics and properties of the membrane. Characterization of the PSf-TiO2 membrane was carried out using FTIR instrumentation to decide on its functional group and SEM to obtain information about the morphology of the membrane surface. In addition, the performance of the PSf-TiO2 membrane was also identified through the acquisition of flux values and percent membrane rejection.

### 3.1 Synthesis and Fabrication of PSf-TiOMembrane

The synthesis process of the PSf-TiO2 membrane begins with making a dope solution consisting of PSf as the primary polymer with a composition of 20%. The dope solution was mixed with 80% N-methyl-2-pyrrolidone (NMP) solvent. The membrane is printed using the phase inversion method, where in this method, it is done by printing the membrane on a glass plate, then flattened using a stainless steel casting knife that is moved down to form a thin layer of the dope solution (Wiley & Sons, 2004) (Fig. 1).

### 3.2 Characterization of PSf-TiO2 Membrane Microscopy Electron Scanning Analysis

One way to determine the morphological structure of the membrane is by Scanning Electron Microscopy (SEM) analysis. SEM analyzed the membrane to decide on the morphological structure of the membrane. The results of the SEM analysis are photos of the surface of the membrane using an electron microscope (Wiley & Sons, 2004). The

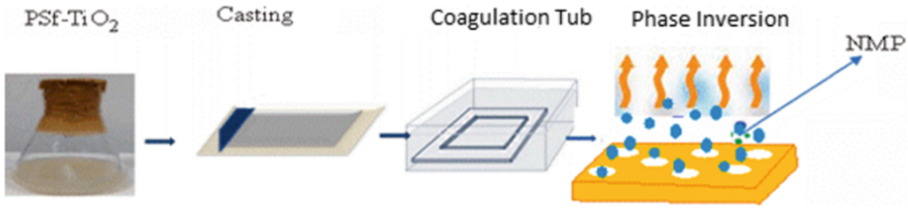


**Fig. 1.** Illustration of PSf-$TiO_2$ Membrane Synthesis Procedure

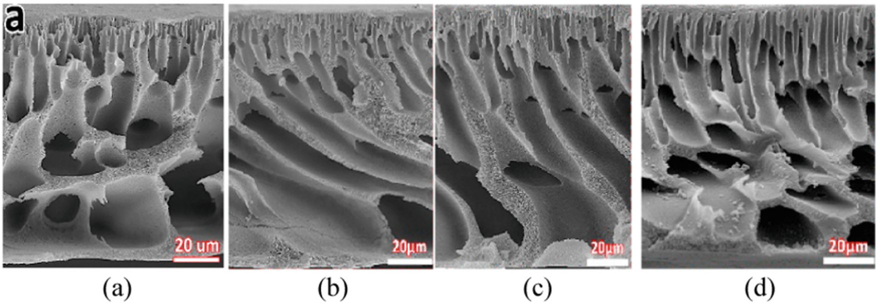


**Fig. 2.** Morphological forms of PSf and PSf-$TiO_2$ membranes via SEM

principle of analysis using SEM is that if there is a change in a material, for example, a change in surface structure, the material tends to experience an energy shift. The changed energy can be emitted, reflected, absorbed and transformed into an electron wave function that can be captured and analyzed (Moeinzadeh et al., 2019). In this research, an asymmetric membrane was produced from a polysulfone polymer using the phase inversion method, as shown by the results of SEM analysis. In the picture, it is clear that there are three layers, namely the dense layer, which is on the top surface of the membrane, which functions as a selective layer, and the intermediate layer, which is the layer between the dense layers. In addition, a buffer layer acts as a mechanical support for the membrane. In this study, morphological analysis was carried out on PSf and PSf-TiO2 membranes.

The SEM results of the membrane cross-section can be seen in Fig. 2 with a magnification of 1000 times. It can be seen in Fig. 2 (a) that the PSf membrane looks clean, in contrast to Fig. 2 (b, c, and d) showing SEM analysis on the surface of the PSF-TiO2 membrane 0.5; 1; and 1.5 wt %., The presence of white spots indicates that the membrane contains TiO2. In addition, the morphological cross-section of the PSf-TiO2 membrane has a cavity shaped like a finger, and the membrane structure looks asymmetric; however, as can be seen, there were no significant differences in the cross-section and morphology of the surface of the PSf-TiO2 membrane due to various variations in %wt.

### 3.3 Fourier Transform Infra Red Analysis

Characterization using FTIR can provide information about the functional groups contained in the membrane as seen from the peaks that appear at specific wavelengths. The results of the description of the PSF-TiO2 membrane using FTIR obtained results, as can be seen in Fig. 3. Characterizing functional groups on membranes using FTIR was carried out in wave numbers 500–4000 $cm^{-1}$.

Membrane characterization using FTIR was used to analyze changes in chemical functional groups during PSf membrane modification. The membrane FTIR spectra are presented in Fig. 3. The FTIR spectra of all membranes have similar transmittance peak patterns, or the differences are barely significant. This indicates that there is no change in the polymer backbone. In addition, adding a certain amount of TiO2 to the PSf membrane is a physical interaction, so it does not affect the bonds formed on the membrane. TiO2 also has a relatively small particle size, which is thought to be well embedded in the PSf membrane (Jyothi et al., 2014). The spectral peaks consistently show a typical polysulfone absorption spectrum. This is indicated by the presence of a mountain at wave number 1240 $cm^{-1}$, which is O=S=O stretching, and the absorbance at 1585 $cm^{-1}$ is defined as the aromatic C=C stretching band of the PSf polymer (Wang et al., 2022). In addition, there is a visible peak at wave number 1600–1400 $cm^{-1}$, which corresponds to the C=C aromatic group from the NMP solvent residue. A C–H group is also marked by a weak peak at wave number 2970 $\pm$ 5 $cm^{-1}$. The PSf membrane before modification showed the highest.

C=C stretching peak at wave number 1584 $cm^{-1}$ with a transmittance value of 81.91%, while a 1.5 wt% PSf-TiO2 membrane showed the lowest transmittance value of 74.27%. The transmittance peak corresponding to the C–H absorption band shows a similar phenomenon. These results indicate that the TiO2 particles have been modified on the membrane, where the TiO2 particles contain –OH.

### 3.4 Contact Angel Angle Analysis

Membrane contact angle measurements were carried out on PSf and PSf-TiO2 membranes with variations of 0.5 wt%, 1wt% and 1.5wt%. Contact angle analysis is used to determine the properties of the membrane to water, whether the membrane that has been synthesized is hydrophobic or hydrophilic, where the membrane is expected to be able to pass water well (Hidayah et al., 2021) from the results obtained, the contact angle values of various variations of TiO2. The PSf membrane has the most significant contact angle value of 61.67. This means that the PSf membrane has more hydrophobic properties compared to PSf-TiO2. At the same time, adding TiO2 to the PSf membrane can increase the hydrophilic properties of the membrane. This means that as the number of TiO2 particles grows, the hydrophilicity also increases while the contact angle decreases, from 59.17 at 0.5 wt% PSf-TiO2 to 51.7 at 1.5 wt% PSf-TiO2 membrane. This is due to TiO2 particles having a large surface area, so TiO2 can easily absorb hydrophobic hydroxyl groups to become hydrophilic. A hydroxyl is a functional group -OH, used as a substituent in an organic compound and is present in the polymer membrane chain (Ahmed et al., 2022). The contact angle test on PSf and PSf-TiO2 membranes can be seen in Fig. 4.

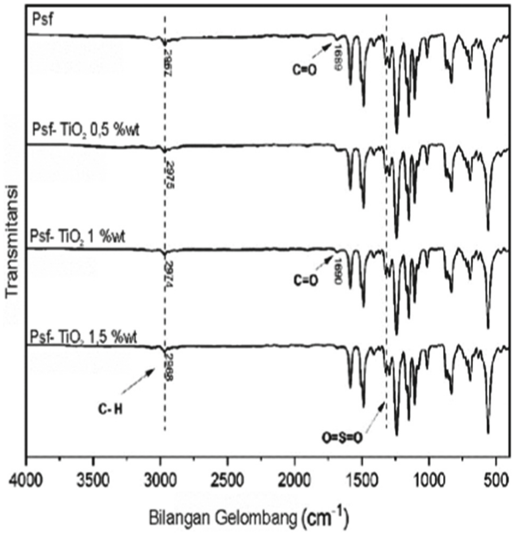


**Fig. 3.** FTIR of PSf and PSf-$TiO_2$ membranes

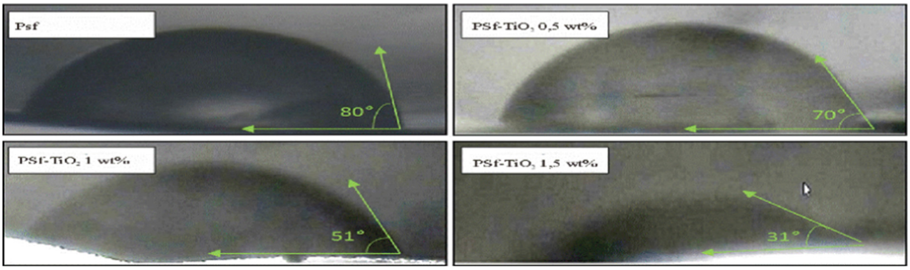


**Fig. 4.** Contact angle test results on PSf and PSf-TiOmembranes 2

### 3.5 Determination of Fe2+ on Levels in Laboratory Waste

Laboratory waste samples were taken from PT. Indo Sea Food. Measurement of Fe2+ ion levels in this study was carried out using an Atomic Absorption Spectrophotometer (AAS). The results of visual observations of laboratory liquid waste have the characteristics of light brown in color, pungent smell and turbid. Wastewater, before being treated, has very high pollutant levels, which can cause the PSf-TiO2 membrane to saturate or damage quickly (Moeinzadeh et al., 2019). So to reduce the pollutant load, the wastewater is diluted first with the aim that the pollutant concentration is reduced so that the processing using PSf-TiO2 membranes can take place more effectively. In this study, laboratory wastewater samples from PT. Indo Sea Food was diluted twice using deionized water with the ratio of wastewater: deionized water being 1:2. The Fe2+ ion content in laboratory wastewater before dilution was 508 ppm, and after dilution, it became 250 ppm.

### 3.6 Effect of TiO2 Concentration on PSf-TiO2 Membrane Performance

The PSF-TiO2 membrane needs evaluation which aims to determine the optimum membrane performance. This test includes the measurement of flux (permeability) and rejection value (selectivity). In this study, the variable concentration of TiO2 added to the 20% PSf membrane was 0.5 wt%, 1 wt% and 1.5 wt%. The difference in the concentration of TiO2 addition to the PSf membrane is expected to show how TiO2 affects the performance of the membrane.

### 3.7 Effect of TiO2 Concentration on Membrane Flux

The addition of TiO2 needs to be seen to determine how it affects membrane performance. One of the membrane performances can be seen from the membrane flux value. Membrane flux measurements were carried out with a dead-end filtration device to measure the membrane flow rate per unit area per unit time. Before measuring the flux, the membrane is compacted using deionized water until the volume that can pass through the membrane is constant in each unit. This is done to strengthen the bonds inside the membrane, so they are not easily torn. In addition, compaction also aims to obtain a constant flux of water (Wang et al., 2022).

After completing the compaction process, PSf and PSf-TiO2 membrane flux was measured every 10 min to 1 h. The area of the membrane used in this study was $10.75 \times 10^{-4}$ $m^2$, with a diameter of 3.70 cm for each membrane. The results of PSf and PSf-TiO2 membrane flux measurements (0.5 wt %, 1 wt %, and 1.5 wt %) can be seen in Table 4.2 and Fig. 4.5. This is because TiO2 can increase the hydrophilicity of the membrane. The hydrophilic nature of TiO2 is proven by the results of the contact angle analysis presented in Table 4.1, where with the addition of TiO2, the contact angle value gets lower. This is because the hydrophilic nature caused by TiO2 makes the Fe2+ ion levels in PT. Indo Sea Food will pass through the membrane more quickly so that the volume of permeate produced will increase, and the flux value will also increase. However, excess TiO2 can cause a lower flux value than the optimum concentration (Peechmani et al., 2021). This can happen because too much TiO2 will cause TiO2 aggregation. TiO2 aggregation can cause fouling on the membrane, where TiO2 will accumulate at one point or not spread as a whole, so the filtration will not be optimal. So it is essential to consider the optimum condition when adding TiO2. This study's optimum conditions occurred on a 0.5 wt% PSf-TiO2 membrane. Based on the flux value data obtained, it can be seen that the PSf membrane and PSf-TiO2 membrane are ultrafiltration membranes that have flux values between 10–50, as shown in Fig. 5.

### 3.8 Effect of TiO2 Concentration on Membrane Rejection

Membrane performance can also be seen from the calculation of the membrane rejection value obtained in the test. The value of membrane rejection can be measured when carrying out the filtration process, together with the measurement of membrane flux. In calculating membrane rejection, what needs to be considered is the permeate concentration after filtration and the concentration before filtration. The rejection measurement is based on the denial of each membrane to Fe2+ ions. The results of measuring the

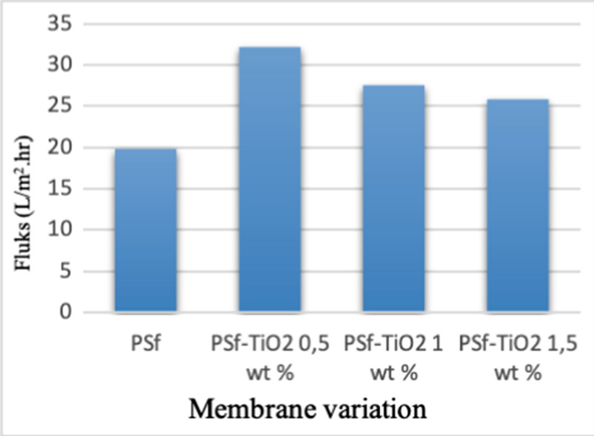


**Fig. 5.** Graph of the Effect of Adding $TiO_2$ on Membrane Flux at different $TiO_2$ Concentration

rejection value of PSf and PSf-TiO2 membranes (0.5 wt %, 1 wt %, and 1.5 wt %) can be seen in Table 4.3 and Fig. 4.6. With the addition of TiO2, the rejection of the membrane increases. This is because adding TiO2 causes the membrane's pores to shrink. So that with a smaller pore, the rejection of the membrane will increase. However, when adding TiO2, you must pay attention to the optimum value because if TiO2 is added inappropriately, it will cause aggregates on the membrane, so the performance of the membrane during the filtration process will decrease or decrease. Aggregates will form on the surface of the membrane that it will make the membrane surface inhomogeneous. This is because an inhomogeneous membrane surface will make the performance of the membrane different on each character so that the ability of the membrane to hold particles on each side of the cover is other (Jyothi et al., 2014).

## 4 Conclusion

In this study, a membrane was created successfully for the filtration of Fe2+ metal ions contained in PT. Indo Sea Food with wet phase inversion method. The conclusions obtained from the study were:

1. Characterization of the contact angle indicates that the PSF-TiO2 membrane is more hydrophilic than the PSF membrane. In contrast, the FTIR characterization shows the presence of a functional group O=S=O at a wave number of 1240 $cm^{-1}$ which indicates the presence of polymer compound content PSf, and the presence in the addition of TiO2 appears the –OH functional group of Ti–OH at wave numbers 1630 and 1600 $cm^{-1}$.
2. The effect of TiO2 addition on membrane performance can increase flux and % membrane rejection on the composition of TiO2 addition by 0.5wt% (PSF-TiO2 0.5wt%).

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