IMPROVEMENT OF PES NANOCOMPOSITE MEMBRANE PERFORMANCE THROUGH UV AND ZnO CONCENTRATION FOR REFINERY WASTE WATER PURIFICATION

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Abstract

Wastewater is generated from the process of purifying large amounts of water. Refinery wastewater can be purified with PES-ZnO nanocomposite membrane technology. Membrane modification to reduce membrane fouling. Therefore, it is important to provide a low-fouling (non-fouling) membrane. However, currently the formation of fouling and clean water quality standards has not been met. Therefore, researchers studied the use of materials that can overcome membrane impurities and an efficient clean water production system. In addition, one alternative to improve membrane performance is to use nanocomposites (ZnO). The membrane was made with a concentration of PES 18 wt% and ZnO 0.5; 1; 1.5% by weight. with the influence of UV light on the membrane for 1,5,10 minutes at a temperature of 180°C. SEM analysis was used to show that the PES membrane has larger pores. Angle contact test was also carried out to show ZnO nanoparticles can improve the hydrophilic properties of membranes. For this reason, a TDS analysis is carried out, namely Cl−, S2− and turbidity. From this analysis shows significant results with membrane performance, modification with UV radiation and the addition of PVA / PEG have interrelated effects to improve membrane performance.

Keywords: Membrane, Fouling, wastewater, Purification, PES-ZnO

I. Introduction

The modern lifestyle depends on a reliable supply of energy. Among the most important sources of energy for mankind are oil, gas, methane, and water which are always produced as by-products which are commonly known as oil refinery wastewater [1]. The wastewater obtained is purified to meet fluid standards when discharged into the river. The use of membrane technology is a solution for oily wastewater that is microns in size [2]. The petroleum industry in Indonesia has increased from year to year. The increase in oil production was offset by the increase in the amount of waste generated from petroleum exploration. This waste contains organic and inorganic materials that have the potential to become B3 waste (Toxic and Hazardous Materials) which affect environmental and human health [3]. So many studies have focused on oil refinery wastewater treatment using membranes. Developments in oil production have led to increased water use and the generation of
wastewater[4]. Several articles on petroleum refinery wastewater treatment have been published, there are several methods to solve the waste water problem such as reverse osmosis filtration, zeolites, surfactants, to membrane filtration. one of the ways to overcome this water crisis is to process oil refinery waste into water that is suitable for use[5]. Improved wastewater treatment technology could be an important part of solving future water crises. Currently available refinery wastewater is limited to re-injection, reuse or recycling which is highly regulated because of the hazard of contaminants to the environment [6]. Traditional methods are no longer able to meet industrial needs, so we need innovative ways to meet industrial needs. Therefore in the petroleum industry it involves refining refined water treatment with a nanofiltration membrane. Membranes are an appropriate technology for water treatment. This technology uses simple operations, without additives, is cost effective, has high productivity. The advantages of this membrane can be used for brackish water, wastewater, desalination of seawater, etc [7].

Polyethersulfone (PES) as a membrane material has superior properties; but it is more semi-hydrophobic, and prone to fouling which limits its practical application [8] However, this hydrophobicity causes more fouling of the membrane [9]. PES membranes can be modified by grafting, coating, and mixing methods. PES has an advantage over coating and grafting because it allows modification of the membrane at the fabrication stage, whereas manufacturing and coating are modifications after fabrication. Mixing is also considered to be the fastest and easiest of these three models [10]. Al-Hinai et al [11] studied fouling on PES Nanocomposite membranes caused by organic materials, such as humic acid, and microorganisms, on the membrane surface. Fouling creates barriers to water in the refinery and wastewater at the factory [12]. Fouling control aims to make membranes better compete with other technologies [13]. To solve the problem of fouling in purification water treatment at the refinery in this study using a polyethersulfone membrane with additive ZnO nanoparticles.

Therefore, this research will develop the use of ZnO nano composites which are expected to reduce agglomeration and fouling. Then the use of ultraviolet will also be developed, where the use of ultraviolet will help to eliminate the interface or defects that occur between polymeric and inorganic membranes. Furthermore, in this study a combination system of polyether sulfone (PES) ultrafiltration membranes will also be developed as the initial processing of produced water, then followed by a polyethersulfone (PES) -nano ZnO nanofiltration membrane. The purpose of using the polyether sulfone (PES) ultrafiltration membrane is as an initial process to remove impurities in the produced water then the process will be refined into clean water on the polyether sulfone (PES) -nano ZnO nanofiltration membrane

2. Material and methods

2.1. Materials

Nano Composit Polyether sulfone (PES) sebagai bahan membran dibeli dari Solvay Advanced Materials (United States of America). Nmethyl-2-pyrrolidone (NMP) was purchased from Merck, USA as a polymer solvent. NanoZnO inorganic additives are supplied by Nano Center Indonesia, Indonesia. Polyvinyl alcohol (PVA) as a surface modifier additive and polyethylene glycol (PEG) 6000 Da and 4000 Da as an aporogenous agent were obtained from Merck, USA. Refinery samples were obtained from petroleum factories (Pertamina, Ltd., Indonesia) with the following initial characteristics: TDS up to 876 mg / L, COD up to 216.5 mg / L, and ammonia up to 45.2 mg / L.
2.2. PES-nano ZnO membrane fabrication

Making PES-ZnO Nano Composite Membranes using a phase inversion model by pouring it on a sheet of glass. The PES-ZnO nano composite solution was prepared by combining 18% PES, 0.5% nano ZnO, and 5% PEG using NMP on the manufacture of polyethersulfone PES-ZnO ultrafiltration membranes, it was started by making a printing solution consisting of PES with an ingredient of 23 wt% in total solids mixed with nano ZnO composition of 0.5 wt%, 1 wt% and 1.5 wt%, with NMP solvent 80 wt% of total solids. Printing on membranes using a dry phase inversion model. this is done by printing the initial membrane on a sheet of glass with a pouring knife. Before soaking in the coagulation place bath, first irradiated with UV for 1 minute, 5 minutes and 10 minutes. Then the glass plate is immersed for a moment in the coagulation bath. The printed membrane is then left for 1 day in clean water. The membranes were then dried at atmospheric temperature for 1 day. Next, do a post thermal annealing treatment on the PES-ZnO nano composite membrane with a temperature of 180°C for 10 seconds. In the next stage, characterization was carried out by determining flux and rejection, Scanning Electron Microscopy (SEM) and contact angle. After that, the membrane application test for produced water treatment is carried out.

2.3. Membrane characterization

2.3.1. the resulting flux and membrane rejection

Result the flux value occurs at concentration is high, the decrease in flux will be faster. To measure the permeability value of the membrane, the filtration cells were placed in the filter [14]. A pressure water test of 8 bar is inserted into the filtration and then closed, Furthermore the compaction process aims to form polymer chains for 30-45 minutes. After compaction, distilled water is replaced with oil refinery wastewater, with flux measurements calculated from the volume of oil waste water on a scale of 40 minutes. Permeate volume is calculated by comparing the flux values [15].

2.3.2. Determination of membrane morphological structure

The results of the morphological structure of the membrane using a Scanning Electron Microscope to analyze the quality of the nanoparticle dispersion [16], by means of The membrane was dried, then immersed in liquid nitrogen until it hardens. Before shooting, the membranes are removed and both ends are cut off. then the membrane pieces are coated with gold (coating) which acts as a conductor. then, membrane sheet photographed at self-determined magnification.

2.4. PES-ZnO membrane application for refinery wastewater purification

Membrane hydrophilicity was measured by measuring the contact angle of the composite membrane using a contact angle measuring instrument (Contact angel). The value of the contact angle for each sample was obtained at four different sample positions and then the average value was recorded [17]. The use of ultraviolet on membranes greatly benefits the performance of ultrafiltration (UF) membranes. for oil refinery wastewater treatment. In this study, UV oxidation was used as a pretreatment for membrane impurity from natural organic matter in surface water [18]. Turbidity of Refinery Wastewater is carried out using a turbidimeter by analyzing the NTU value before and after passing through the membrane. Membrane filtration was performed for 2.5 hours with a crossflow system, using a membrane holder with an active area of 12.57 cm² and an absolute pressure of 6 bar. The performance of the membrane was based on flux and rejection.
2.4.1. Permeate Flux is calculated by Equation 1:

\[ J_w = \frac{V}{A \cdot t} \]  

\( J_w \) is the flux (L/m\(^2\)·h), \( V \) is the permeates volume (L), \( A \) is the active membrane area (12.57 cm\(^2\)), and \( t \) is the operating time interval (hour).

2.4.2. The rejections is calculated by equation 2:

\[ R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \]  

where \( C_p \) (mg/L) and \( C_f \) (mg/L) are concentration. Rejection is calculated based on the reduction of contaminants in the permeate which then is evaluated. In this study, the studied wastewater parameter TDS is analyzed using a TDS KIT (HM original), and ammonia is analyzed using a UV spectrophotometer (Shimadzu BioSpec-mini UV-Vis Spectrophotometer).

2.4.3. The flux reduction ratio is calculated by equation 3:

\[ M\% = \left(\frac{J_0 - J_1}{J_0}\right) \times 100\% \]  

\( J_0 \) (L/m\(^2\)·h\(^{-1}\)) is the pure water flux, and \( J_1 \) (L/m\(^2\)·h\(^{-1}\)) is the flux of wastewater during filtration at 150 minutes.

3. Results and discussions

3.1. Characterization of Making Membrane Composites

3.1.1. Scanning Electron Microscopy Analysis

The structure of the membrane surface and its cross section were analyzed by SEM. This analysis provides qualitative information regarding membrane pore size, pore distribution and overall pore geometry. The membrane is immersed in a liquid nitrogen solution so that the membrane is easily broken then it is attached to the sample container (brass disk) with the help of tape. This membrane sample is coated with gold in a vacuum. After that the membrane layer can be seen with an electron microscope and photographed. The results of the Scanning Electron Microscopy (SEM) analysis are surface and cross-sectional photographs of the membrane using an electron microscope [19]. There are several membrane characterizations tested in this study, including membrane I (PES 18 wt%), membrane II (PES 18 wt% 0.5 wt% ZnO), and membrane III (PES 18 wt% 0.5% ZnO-UV 10 minutes and thermal annealing at 180°C).

Figure 3.1 is a picture of SEM analysis results on the 18 wt% PES membrane. In Figure 3.1, it can be seen that the surface is almost clean, in contrast to Figure 3.2. Figure 3.2 shows SEM analysis of the surface and transverse PES membranes of 18 wt% 0.5 wt% ZnO. In Figure 3.2, there are white spots indicating that the membrane contains ZnO nanoparticles. Meanwhile, for cross section analysis on both membranes, it can be seen that the resulting membrane is asymmetrical. Because the results of the analysis form more than 3 membrane layers. namely the dense layer that is Figure 3.1 is a picture of SEM analysis results on the 18 wt% PES membrane.
Figure 3.1 SEM analysis of the surface and transverse 18 wt% PES membrane

Figure 3.2 SEM analysis of surface and transverse PES membranes 18 wt% 0.5 wt% ZnO

Figure 3.2 shows the results of SEM analysis on membranes and transverse, PES membranes 18 wt% 0.5 wt% ZnO without thermal annealing and UV, while Figure 3.3 shows SEM analysis in the presence of thermal annealing and UV. In Figure 3.3b, it is very clear that the full UV and thermal annealing causes the pores membrane to be slightly larger and more uniform than in Figure 3.2b. These larger pores cause the performance of the membrane on all surfaces to be the same. So that when filtering or filtration it will increase the flux and rejection value of the membrane. The longer the solution passes through the membrane, the smaller the flux value [19]. This is because fouling occurs deposition of suspended solids or dissolved solids on the membrane surface or in the pores of the membrane, resulting in a decrease in membrane performance [20]. Figure 3.3 is also an asymmetric membrane, because the results of the analysis show the dense layer, intermediate layer, and porous substructure.
In Figure 3.3 it can be seen in the surface analysis of the causes of fouling is caused by chemical materials such as organic and inorganic compounds as well as biological materials such as microorganisms. Fouling causes the thermal flux to fall, so frequent membrane replacement and washing are required [21]. Since membrane fouling is a consequence of the interactions between the membrane surface and the solute by different mechanisms and hydrophobic interactions are usually accepted as dominant for PES membranes, the surface tension dispersion component can be a good predictor of fouling [22]. Fouling has the greatest influence on the operation of the filtration installation. This is because fouling is a very complex phenomenon involving many factors. However, the influence of ZnO on the membrane can reduce the process of fouling formation.

3.1.2. Water Contact angle analysis

Contact angle measurement is used to indicate whether the membrane is hydrophilic or hydrophobic. Where the hydrophilic nature means that the compound "likes water" is able to pass water well which has a contact angle below 90°, while hydrophobic "doesn't like water" which has a contact angle value greater than 90° which indicates that the hydrophobic surface is difficult to pass water at the time. filtration process. Contact angle measurement is considered important at this time because it can be used as a way of knowing the phenomenon of wettability [23]. Tables 3.1 and 3.2 show the results of the contact angle measurements of each membrane.

| No | Membrane | Membran Composition                          | Contact angle (°) |
|----|----------|---------------------------------------------|-------------------|
| 1  | PES 1    | PES 18%                                     | 66                |
| 2  | PES 2    | PES 18%+0,5NanoZnO+1UV                      | 64,24             |
| 3  | PES 3    | PES 18%+1NanoZnO+1UV                        | 58,50             |
| 4  | PES 4    | PES 18%+1,5NanoZnO+1UV                      | 56,63             |

| No | Membrane | Membran Composition                          | Contact angle (°) |
|----|----------|---------------------------------------------|-------------------|
| 1  | PES 5    | PES 18% + 0,5NanoZnO + withoutUV            | 65                |
| 2  | PES 6    | PES 18%+0,5NanoZnO +1UV                     | 68,24             |
Based on Tables 3.1 and 3.2, get the value of contact angle on PES, PES with ZnO nanoparticles with variations of 0.5% wt, 1% wt and 1.5% wt and also variations in UV irradiation on membranes with irradiation times of 1 minute, 5 minutes and 10 minutes. PES without nanoparticles and UV has the greatest contact angle value, which is 66°. From the results of this study PES is hydrophobic, while the presence of ZnO can increase the hydrophilic properties of the membrane, namely by decreasing the contact angle from 64.24° to 56.63°. The contact angle is an important parameter for seeing the surface of an object that is hydrophobic or hydrophilic. Hydrophobic properties are waterproof and have a contact angle between 90° - 150°, while hydrophobic properties have a contact angle between 10° - 90° [24].

The contact angle is also a determining factor for the wetting of a surface. Measurement of the digital image processing point of view usually uses a circle shape. UV irradiation can increase the surface hydrophilicity of the membrane, according to Table 3.1 - 3.2. This is due to the formation of hydrophilic functional groups in PES, to form the free radical molecule PES [23].

3.2. Separation Performance Using A Membrane
3.2.1. Effect of Ultra Violet Irradiation on Membranes

UV rays here function as free radical formers that will bind to other monomers. In this case PES and PEG will both form free radicals and the two polymers will become charged so that they will bind together to form a bond between PES and PEG (polymerization mechanism) [25]. With this bond, not only does PEG mix, but also chemical bonds occur which make the membrane more stable and more resistant to fouling. To see how ultraviolet (UV) affects membrane performance, several variables are made. UV irradiation was carried out in 1, 5, and 10 minutes for each variable PES ZnO 0.5wt%, PES ZnO 1wt%, and 1.5wt%. Membrane performance after modification was seen from the flux value and its rejection value.

3.2.2. Effect of UV Irradiation on Membrane Flux

![Figure 3.4](image_url)

This figure shows the effect of UV irradiation time, namely 1 minute, 5 minutes and 10 minutes on the flux value at 0.5% by weight PES Nano ZnO membrane. The membrane flux decreases with the length of the filtration time, compared to the flux in a membrane that does not use UV light. This is the time difference between the membrane before and after printing which is inserted into the coagulation bath.
so that it makes the membrane non-porous [18]. This event is due to the membrane that is exposed to UV light, there will be a cutting of the polymer chain (chain scission) and the process of cross-linking. UV spectroscopy shows the visible light absorption ability of the nano composite membrane due to the presence of nitrogen as a dopant in the membrane [26]. UV-based advanced oxidation processes have become a promising strategy for drinking water treatment due to the strong oxidative radicals generated.

The highest flux value was in PES \( \text{nanoZnO 0.5\% wt} \) with 10 minutes of irradiation. In the initial state, the membrane flux value was 30,886 Lh\(^{-1}\)m\(^{-2}\) then decreased to 7.20 Lh\(^{-1}\)m\(^{-2}\). Meanwhile, the lowest flux value was in the PES-ZnO membrane 0.5\% wt without UV irradiation. The membrane without UV light added by PEG has a greater flux than the membrane without PEG because PEG has a function as a membrane pore-forming so that it can produce more pores and a large membrane flux. However, after being exposed to UV light, the membrane flux with PEG decreased even under the membrane flux without PEG. This is because the PES membrane undergoes degradation which causes its flux to continue to increase.

3.2.3. Effect of Ultra Violet Irradiation on Membrane Rejection

Membrane needs to be tested for rejection. The results of filtration or permeate are taken every minute to 40, 80, 140 and 160, then analyzed the results of TDS, \( \text{SO}_4^{2-} \) levels, \( \text{Cl}^- \) levels and turbidity or turbidity so that each percent of the rejection is obtained. TDS stands for Total Dissolved Solid (Amount of Dissolved Metal Solids in Water). TDS itself is a parameter of the number of particles both organic and non-organic. The solutes that are usually present in water itself are usually sodium, magnesium, potassium, manganese, chloride, sulfate, nitrate and many others. The higher the TDS value, the more dissolved metal content in the water the body drinks. Due to the high TDS value in oil wastewater, water recovery by membrane separation can be energy intensive [27]. TDS measurements were carried out with a TDS meter while the \( \text{SO}_4^{2-} \) and \( \text{Cl}^- \) ion analysis on the permeate was carried out using titration. For turbidity using a turbidi meter.

![Figure 3.5 Effect of Ultraviolet irradiation on TDS rejection](image-url)
Figure 3.6. Effect of Ultraviolet irradiation on S2 rejection

Figure 3.7. The Effect of Ultraviolet Irradiation on Cl\textsuperscript{-} Rejection

Figure 3.8. The Effect of Ultraviolet Irradiation on Turbidity Rejection

Figure 3.5 shows the effect of UV irradiation on the percent rejection of TDS. Based on this figure, From this study, the filtration time was obtained, the higher the rejection value[28]. However, the longer the UV exposure, the percentage of rejection decreases. The rejection rate for the TDS parameter according to the study, which is in the range of 9.38-18.75% depending on the feed concentration, pressure, pH, and temperature. The percentage of rejection of S\textsubscript{2} ion. Figure 3.6. It can
be seen the phenomenon in TDS rejection is also experienced by S\textsubscript{2} ion rejection - according to the research, namely in the range of 13.66-48.29% the longer the filtering of rejection values increases but the longer it is done. UV irradiation, then the percent rejection has decreased. Based on Figure 3.7. The rejection rate for parameter Cl\textsuperscript{-} according to the study was in the range of 3.3 - 32 minutes. This rejection rate decreased with increasing UV exposure time. However, it increases with the filtration time. The rate of chloride ion rejection increases with increasing operating pressure because the rate of water transfer across the membrane is faster than the transfer rate of chloride ions [29]. The percent rejection of turbidity is shown in Figure 3.8. Turbidity has Nephelometric Turbidity Units (NTUs) as the standard unit for measuring turbidity. What happens is that Ultraviolet radiation can stop the polymer chain [30].

3.3. Effect of ZnO Concentration on Membrane Performance

Apart from wanting to see the effect of UV irradiation, a variable concentration of ZnO was also treated at 0,5wt%, 1 wt% and 1,5wt% mixed/added with PES 18 wt%. With the different concentrations of ZnO made, it is expected to show how ZnO affects membrane performance.

3.3.1. Effect of ZnO Concentration on Membrane Flux

To determine the effect of ZnO addition, it can be seen from the membrane performance. one of them is flux. The flux value is obtained by comparing the flow rate per unit area per unit time per unit pressure.

![Figure 3.9 Effect of ZnO Concentration on Flux](image)

From Figure 3.9 it can be seen that the flux decreases with increasing concentration of acetone (5-20% by weight)[31]. The longer the filtration, the higher the pressure. and causes fouling on the membrane so that the membrane works properly because it is able to filter out particulates. The longer the use of the membrane the more dirt causes the membrane pores to be closed. This is what makes the pressure rise, the membrane works heavier and results in a decrease in the volume of permeate produced. The decrease in volume causes a decrease in the flux value, because the flux value is directly proportional to the permeate volume. In addition, there is a difference in the flux value at the ZnO concentration, where the flux with the addition of ZnO is higher than that without ZnO.
3.3.2. Effect of ZnO Concentration on Membrane Rejection

The membrane work is seen from the membrane rejection value during the filtration process, together with the measurement of flux. In calculating membrane rejection, what needs to be seen is the permeate concentration after filtration and the concentration of produced water before filtration. Measurement of the rejection of each membrane to ions was seen from the TDS, turbidity, S₂⁻ and Cl⁻ values. In Figure 3.10. It is seen that the addition of ZnO membrane rejection will increase because the pores of the membrane shrink. So that membrane rejection will increase [32]

![Figure 3.10. Results of the Effect of PES-ZnO Concentration on TDS Rejection](image)

![Figure 3.11. Result of the Effect of PES-ZnO Concentration on S2 Rejection](image)

![Figure 3.12. Result of the Effect of PES- ZnO Concentration on Cl- Rejection](image)
Figure 3.11 showed the effect of ZnO concentration on the percent rejection of TDS. Based on this research, the longer the filtration time of the membrane rejection value. However, the greater the ZnO concentration, the lower the percent rejection. While S2 ion rejection is shown in Figure 3.11, S2 rejection - the longer the filtering, the higher the rejection value. The same thing happened to the Cl-rejection, shown in Figure 3.13. Meanwhile, Figure 3.14 shows the turbidity rejection.

4. Conclusion
Repair of nano composite pes-zno membranes through uv and ZnO concentrations for refinery wastewater purification was successfully carried out. SEM results show the formation of an asymmetric membrane with a porous membrane layer structure. The addition of ZnO nanoparticles and coating the membrane using PEG significantly decreased the air contact angle, but the ZnO concentration to a higher concentration indicated an increase in the contact angle. The flux obtained on the membrane with the highest flux value at PES nano-ZnO 0.5% wt with an irradiation time of 10 minutes, the longer the filtration time the higher the membrane rejection value. However, the longer the percentage of UV rejection exposure decreased the rate of return for the TDS parameter according to the study, namely in the range of 9.38-18.75% depending on the concentration of feed. So that the separation can be increased.

Recognition
We thank you for the facility support from the Chemical Engineering waste management laboratory

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