Synthesis and Characterization of Polyacrilonitrile-Fe2o3 Membrane Nano Composites for Oil Refinery Liquid Waste Separation

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ABSTRACT
Synthesis of nano-composite membranes using a phase inversion technique with polymer polyacrilonitrile-fe2o3 membrane for the separation of oil refinery effluent by coating using crosslinked PVA obtained results about the effect of the concentration of nano-composite polyacrilonitrile-Fe2O3 membrane on membrane performance can increase flux and rejection because Fe2O3 has properties that can increase hydrophilic properties. The effect of the duration of ultra violet irradiation on the performance of nano-composite polyacrilonitrile-Fe2O3 membrane for the separation of oil refinery wastewater, makes the membrane flux value increase but the rejection value decreases. The addition of PEG to increase the work and characterization of nano-composite membranes in the separation of oil refinery waste increases. Significant results can be seen from SEM, fourier transform electron microscopy (FTIR) and mechanical tests on membranes.

Keywords : *polyacrilonitrile, PEG, FTIR, Flux, rejection and mechanical membrane.*

1. INTRODUCTION

Petroleum is the main energy source used in households, industry and transportation. This has led to increased exploration, exploitation, processing and transportation of petroleum production activities to meet human needs, thereby increasing the tendency to pollute the environment, especially in coastal areas. The pollution comes from oil refinery waste, by-products from the production, distribution and transportation processes. Waste generated from oil refineries is in the form of liquid waste and solid waste [1]. Oil refinery production of 1000 barrels per day will produce solid waste (oil mud) of more than 2.6 barrels while in Indonesia, refinery production produces crude oil of around 1.2 million barrels per day which means it produces solid waste of 3120 barrels per day and in One year, it produces 1.3 million barrels of waste, of which 285,000 barrels are B3 (Hazardous and Toxic Materials) waste[2]. Petroleum mud waste affects coastal ecosystems, both coral reefs, mangroves and aquatic biota, both lethal (deadly) and sublethal. (inhibit growth, reproduction and other physiological processes)[3]. This is because of the hydrocarbon compounds contained in crude oil [4], which have complex components, including Benzene, Toluene, Ethilbenzene and Xylene isomer (BTEX), which are aromatic compounds in small amounts in hydrocarbons [5]. Extensive oil exploration has caused environmental problems for many years. In addition to oil spills, insufficient treatment of oil refinery wastewater and dumped waste are other potential sources of pollution that have not received sufficient attention from the government and the public [6]. By reviewing current oil refinery wastewater treatment processes and effluent disposal standards, raise awareness.

2. MATERIALS AND METHODS

Materials used

The materials used in this study include: polyacrylonitrile (PAN), Fe2O3, n-methyl-pyrrolidone (NMP), oil refinery wastewater,

Tools used

The tools used in this research are turbidimeter, magnetic stirrer, glass plate, coagulation bath, casting knife, tape, oven, SEM equipment, FTIR equipment, titration equipment, spectrophotometric equipment, and dead end filtration cells.

Polyacrylonitrile (PAN) Membrane Synthesis

The manufacture of membranes made from polyacrylonitrile polymer (PAN) goes through two stages, the first stage is membrane characterization, and the membrane application stage for oil refinery wastewater treatment. At the stage of making polyacrylonitrile membranes (starting with making a printing solution consisting of polyacrylonitrile as a polymer with a composition of 18% wt. Addition of 1, 0.5, and 1% wt nano Fe2O3. Polyethylene glycol (PEG) 6000 as additive with a composition of 2.5, 3.5, and 5%wt for polyacrylonitrile membranes, the remainder as solvent n-methyl-2-pyrrolidone (NMP).The membrane printing uses the phase inversion method. This method is carried out by printing the membrane on a glass plate using a casting knife, then the glass plate is immersed in a bath. coagulation. This research is looking for the effect of post-treatment, namely temperature. The membrane with the best performance is then heated to its optimum temperature with a heating time of 10, 20 and 30 seconds for polyacrylonitrile. The addition of UV irradiation 1, 3, 5 minutes. Finally, the addition of polyvinyl alcohol with concentration of 1, 2, 3% wt. The next step is characterization by determining flux and rejection, scanning electron microscopy (SEM), co ntak angel, fourier transform electron microscopy (FTIR) and mechanical testing. After the membrane application test for oil refinery wastewater treatment was carried out.drawbacks of current wastewater treatment technologies and to discuss the consequences of inadequate treatment on the environment. The use of membrane nanotechnology as an alternative technology that can purify oil refinery waste [7]. One alternative technology that can be used for the treatment of produced water is membrane technology which can be used as a new water source for agricultural irrigation, industrial air, and drinking water. Therefore, an effective technology is needed for the treatment of produced water.

Oil refinery waste treatment using the principle of gravity and using oxidation and coagulation processes. Technology use it supports to produce output according to the disposal standards, however the other hand produces sludge which includes waste (hazardous and toxic materials) with quite a lot of volume [8]. Technology membranes, particularly microfiltration and ultrafiltration, is an alternative method for separate the oil from the air [9]. This technology has high efficiency in separating oil, no requires chemicals in the process to produce air with consistent quality and also this technology can treat waste oil in water by particles less than 5 microns, which is not can be treated with a hydrocyclone [10]. Membranes have been used to treat The feed is in the form of an oil emulsion such as crude oil emulsion, kerosene oil emulsion and crude oil [11]. Results from research in above mentioned that the average membrane can rejects oil, COD and surfactants above 90% [12]. Researches for waste treatment desire in the form of an oil-water emulsion with UF membranes generally use the original waste containing complex ingredients. The parameters in waste components that affect flux permeate cannot be clearly identified. On In this study, the emulsion waste model feed was used oil with the dispersed phase in the form of oil lubricants, gasoline and diesel. The continuous phase is air, while the surfactant is a mediator. Oil lubricants, gasoline and diesel are used to represent petroleum refinery waste originating from the distillation column as well as waste from equipment operation process in a utility plant. This research is related on membrane performance testing (flux and rejection) oil-water emulsion ultrafiltration. With models oil-water waste which is simpler then specific effect of oil on membrane performance can be known

Composite nanomembrane is one of the most widely applied active ingredients in water treatment systems because it uses a low pressure process so that it requires less energy and is economical in manufacturing. [13] and has a higher flux yield compared to other membranes [14]. Therefore [15] stated that one of the most widely used polymers in membrane processes is polyacrylonitrile which can be dissolved in many ventilated polar organic solvents such as DMAc, DMF and NMP. The advantage of polyacrylonitrile is to use the phase inversion method. Coagulation temperature and concentration cause the membrane pore size to enlarge as well as increase in casting solution concentration can cause a decrease in pore size., During the phase inversion process both thermodynamic and this research will develop a nano-composite polyacrylonitrile-Fe2O3 membrane to improve membrane performance in oil refinery wastewater treatment. Nano-composite membrane developed with a novelty in this research is anti-fouling properties which were developed by modifying the nano-composite polyacrylonitrile-Fe2O3 membrane using UV light. One of the purposes of using the addition of nano Fe2O3 is to reduce fouling between polymers and inorganic materials. In addition, it will be able to reduce agglomeration on the nano-composite membrane material. Inorganic membranes that will be used include polyacrylonitrile process will be carried out or combined with post-treatment. The pre-treatment was carried out using ultraviolet light while the post-treatment was carried out using the FTIR test, thickness test and membrane tensile test.

3. RESULT AND DISCUSSION

3.1. Scanning Electron Microscopy (SEM) Analysis

Surface and cross-sectional structure across the membrane were analyzed by SEM. This analysis provides qualitative information regarding membrane pore size, distribution pores and overall pore geometry [16]. The membrane is immersed in a nitrogen solution liquid so that the membrane is easily broken then affixed to the sample container (brass disk) with the help of tape. This membrane footage is plated with gold in a vacuum. After that membrane surface can be observed through electron microscopy and take a photo Figure 3.1 A and B are the results SEM photo analysis to see morphology membrane based on surface membrane (outer surface) and structure transverse membrane (cross section analysis result of 18% wt PAN membrane. In Figure 3.1A it looks clean and 3.1.B shows SEM analysis on the surface and transverse PAN 19 wt % 0.5 wt % Fe2O3 membrane.

Figure 3.1B shows that the pores of the membrane are slightly larger and more uniform. As seen in the cross section analysis of nano composite membranes, the presence of fouling occurs because there are inorganic chemicals (organic/) and microorganisms. With the presence of Fouling flux becomes down, membrane replacement/washing is carried out [17].

3.2. Effect of Fe2O3 Concentration on Membrane Flux

To determine the effect of the addition of Fe2O3 can be seen from the value of the membrane flux. The flux value of a membrane can be calculated by comparing the membrane permeate flow rate per unit area per unit time per unit pressure.

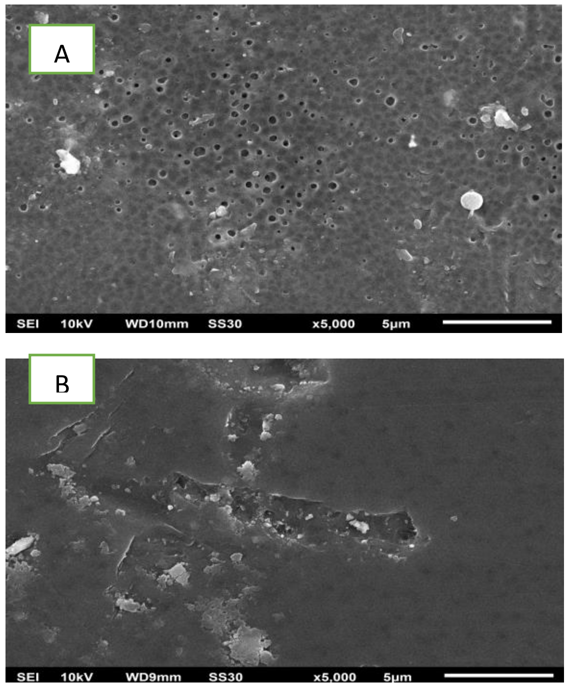

Figure 3.1. SEM analysis on nano composite membrane PAN 18 wt %

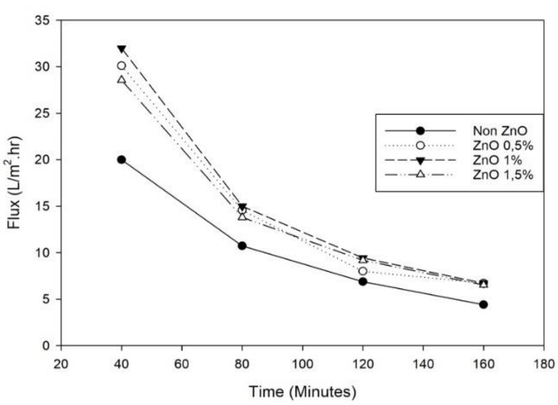

Figure 3.2. Effect of Fe2O3 Concentration on Flux

Figure 3.2. seen flux decreased with increasing concentration of acetone (5-20 wt%)[18]. The longer the filtration time, the pressure of the filtration process will also increase. The increase in pressure indicates the presence of fouling on the membrane surface. This fouling also shows that the membrane works well because it is able to filter out particulates, so that they will accumulate on the membrane surface. The longer the use of the membrane, the dirt on the surface of the membrane will also increase, which can cause the membrane pores to close. This causes the pressure to increase, the work of the membrane becomes heavier and results in a decrease in the volume of permeate produced. The resulting decrease in volume causes a decrease in the flux value as well, because the flux value is directly proportional to the volume of permeate produced for a certain time. Apart from the decrease in flux, it can also be seen from Figure 3.2. that there is a difference in the flux value of each Fe2O3 concentration, where the flux value added with Fe2O3 is higher than without Fe2O3. This is because Fe2O3 can increase the hydrophilicity of the membrane.

3.3. Effect of Fe2O3 Concentration on Membrane Rejection

The performance of the membrane can be seen in the rejection value of the membrane. In calculating membrane rejection, what needs to be seen is the concentration of permeate after filtration and the concentration of oil refinery wastewater before and after filtration.. The rejection measurement is based on the rejection of each membrane to TDS.

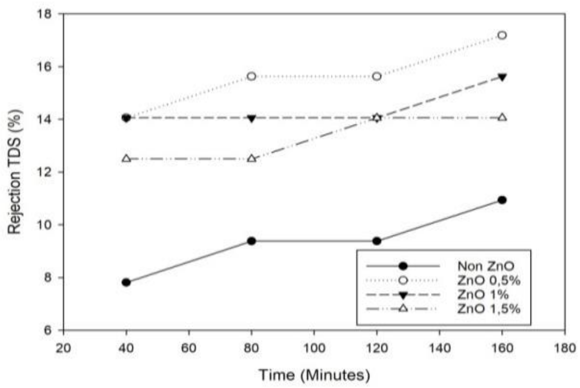

Figure 3.3. Effect of Fe2O3 Concentration on TDS . Rejection

In Figure 3.3. it can be seen that with the addition of Fe2O3 the rejection from the membrane will increase. This is because the addition of Fe2O3 causes the pores of the membrane to shrink. So with smaller pores, the ejection from the membrane will increase [19]. Figure 3.3. showed the effect of Fe2O3 concentration on the percentage of TDS rejection. Based on the figure, it can be seen that the longer the filtration time, the higher the rejection value of the membrane. However, the greater the Fe2O3 concentration, the lower the rejection percentage.

3.4. Effect of Ultraviolet (UV) Radiation on Membrane Performance

UV function to form free radicals with other monomers. In addition of additives PAN and PEG will combine to become free radicals and charged and bonding occurs [20]. With this bond, PEG produces a membrane that is stable and resistant to fouling. To see how the influence of ultraviolet (UV) on the performance of the membrane, several variables were made. UV irradiation was carried out in 1, 5, and 10 minutes on each variable PAN-Fe2O3 0.5wt%, 1wt%, and 1.5wt%. The performance of the membrane after modification is seen from the flux value and rejection value. flux nano composite membrane with UV irradiation for 5 minutes. This is due to the time span between the nano-composite membrane after being molded and before being put into the bath which causes the formation of a non-porous membrane [11]. [21] Ultraviolet (UV) irradiation causes chain cutting and cross-linking. UV-spectroscopy confirmed that the highly visible light absorption ability of the nano composite membrane was due to the presence of nitrogen as a dopant in the membrane structure [22].

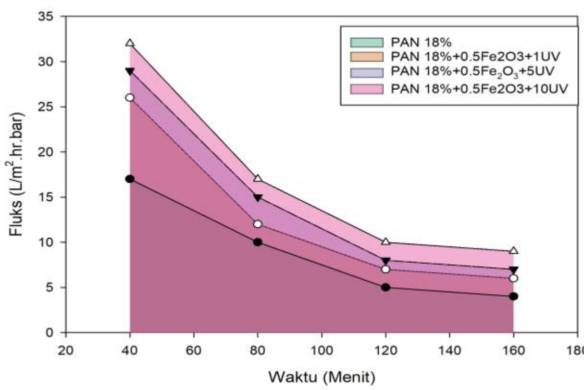

Figure 3.4. Effect of UV irradiation on the value of Flux

In Figure 3.4. UV-based advanced oxidation processes have become a promising strategy for sewage treatment. The highest flux value was in PAN-Fe2O3 0.5%wt with 10 minutes of irradiation. In the initial state, the membrane flux value was 30,886 Lh-1m-2 and then decreased to 7.20 Lh-1m-2. Meanwhile, the lowest flux value was in 0.5%wt PAN-Fe2O3 membrane without UV irradiation. Nano composite membrane with the addition of PEG without ultraviolet light irradiation flux value is greater than the nano composite membrane without PEG due to more membrane pores. However, the flux of the membrane added with PEG decreased after ultraviolet irradiation, this was due to degradation. The polymer chain cutting process occurs at low temperatures, but the crosslinking process occurs at high temperatures [23]. The polymer chain that occurs in the PAN-PEG bond causes the pores to shrink and the flux decreases.

3.5. Effect of UV Radiation on Membrane Rejection

In knowing the performance of nano-composite membranes, it can be seen from the rejection percent. The most commonly used method of determining total dissolve solvent (TDS) in water supplies is specific conductivity measurement with a conductivity probe that detects the presence of ions in water. The conductivity measurements are converted to TDS values using a factor that varies according to the type of water . The practical quantity limit for TDS in water by this method is 10 mg/litre[24]. High concentrations of TDS can also be measured gravimetrically, although volatile organic compounds are lost by this method. The TDS constituents can also be measured individually. Total dissolved solids (TDS) is a term used to describe inorganic salts and small amounts of organic matter present in aqueous solution [24].

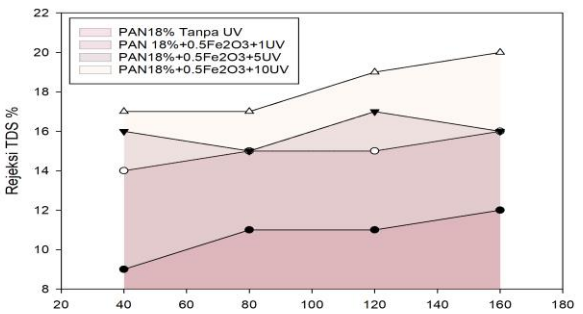

Figure 3.5. showed the effect of UV irradiation on the percentage of TDS rejection.

Based on the figure, it can be seen that the longer the filtration or filtration time, the higher the rejection value of the membrane. However, the longer the UV irradiation the percentage of rejection decreased. The rejection rate for the TDS parameter was in accordance with the study, which was in the range of 9.38-18.75% depending on the feed concentration, pressure, pH, and temperature. This phenomenon is caused by the effect of UV irradiation causing the membrane to undergo chain scission and crosslinking which results in a denser membrane pore (denser) thereby preventing more contaminants from escaping to the permeate. An increase in pure water flux was observed after only a few minutes of UV exposure in aqueous media. The pure water flux doubled with 10 min UV irradiation, and a sixfold increase in pure water flux occurred after 20 min irradiation[25]. So that the membrane pores get bigger and make it easier for water to diffuse through the membrane pores. However, the longer the filtration time, the overall percent ion rejection increased. This is because inorganic and organic substances in the oil refinery wastewater have been retained on the membrane surface.

3.6. Effect of Fe_2O_3 Concentration on Membrane Flux

To determine the effect of the addition of Fe2O3, the flux and rejection performance of the membrane can be analyzed. One of the membrane performances can be seen from the membrane flux value. The flux value of a membrane can be calculated by comparing the membrane permeate flow rate per unit area per unit time per unit pressure. From Figure 3.6. seen flux decreased with increasing concentration of acetone (5-20 wt%)[18]. The longer the filtration time, the pressure of the filtration process will also increase. The longer the use of the membrane, the more dirt on the surface of the membrane, so that the pores of the membrane are closed.. This causes the pressure to increase, the work of the membrane becomes heavier and results in a decrease in the volume of permeate produced. The resulting decrease in volume causes a decrease in the flux value as well, because the flux value is directly proportional to the volume of permeate produced for a certain time.

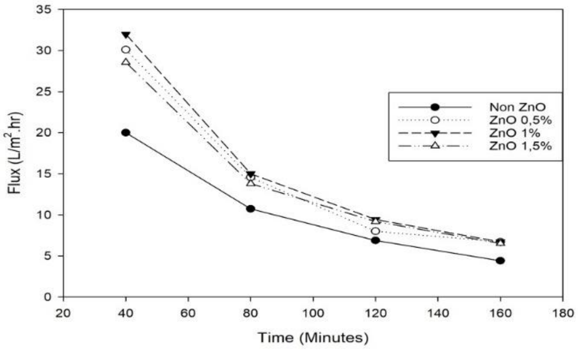

Figure 3.6. Effect of Fe_2O_3 Concentration on Flux

Apart from the decrease in flux, it can also be seen from Figure 3.6. that there is a difference in flux values for Fe_2O_3 from each concentration of Fe_2O_3, where the flux value added with $Fe_2O_3$is higher than without Fe_2O_3. This is because Fe_2O_3 can increase the hydrophilicity of the membrane.

3.7. Effect of Fe_2O_3 Concentration on Membrane Rejection

The performance of the membrane can also be seen in the rejection value of the membrane. The membrane rejection value can be measured during the filtration process, along with the membrane flux measurement. In calculating the membrane rejection, what needs to be seen is the concentration of permeate after filtration and the concentration of oil refinery wastewater before filtration. The rejection measurement is based on the rejection of each membrane to TDS.

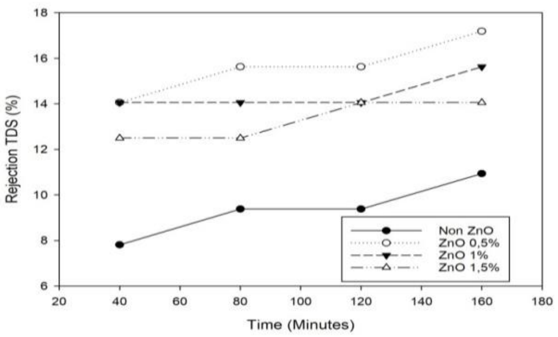

Figure 3.7. Effect of Fe2O3 Concentration on TDS . Rejection

In Figure 3.7. it can be seen that with the addition of Fe2O3 the rejection from the membrane will increase. This is because the addition of Fe2O3 causes the pores of the membrane to shrink. So with smaller pores, the ejection from the membrane will increase [19].

Figure 3.7. showed the effect of Fe2O3 concentration on the percentage of TDS rejection. Based on the figure, it can be seen that the longer the filtration time, the higher the rejection value of the membrane. However, the greater the Fe2O3 concentration, the lower the rejection percentage.

3.8. Thickness Test

Thickness testing is a non-destructive test method carried out using a thickness gauge to determine the total thickness of the installed waterproof membrane [26]. Membranes with various concentrations of PEG tend to increase the thickness of the membrane. From the graph, it can be seen that the highest nanocomposite membrane thickness was 0.028 mm at 3% PEG concentration and the lowest was 0.017 mm at 9% PEG concentration. This membrane is thinner when compared to the gelatinous polymer membrane [27]. The thickness of the membrane affects the characterization of the nano composite membrane [28]. The addition of PEG concentration can be seen in Figure 3.8

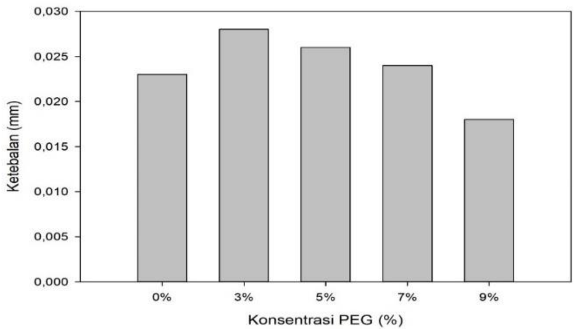

Figure 3.8. Graph of the relationship between PEG concentration and membrane thickness

3.9. Membrane Porosity Test

The porosity test (swelling) is carried out to determine the amount of substance that can be absorbed by the membrane. Porosity is based on the law of capillary equilibrium which measures the equilibrium distribution of the working fluid between the sample and the standard (which has a known porometric curve). If the pore size distribution of one of the porous bodies is known (i.e., the standard), then the pore size distribution of the other body (i.e., the sample) can be calculated by determining the distribution of the liquid between all the porous bodies.

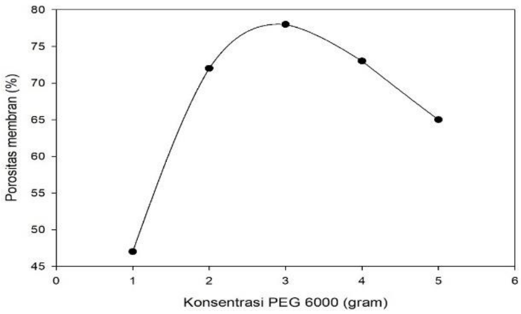

Figure 3.9. Relationship Between PEG 6000 Concentration and Membrane Porosity

The porosity test in this study was carried out on water. Equilibrium water content is one method of membrane characterization to determine the degree of hydrophilicity of the membrane, which is related to water flux and membrane porosity. the results can be seen in Figure 3.9Figure 3.9. shows the results of the PAN membrane porosity test. The porosity of the membrane is calculated using the average mass of the polymer material and the mass of the liquid in the membrane. The membrane volume was determined gravimetrically by weighing a sample of known area and thickness. The density of the membrane polymer material was estimated from the literature. The thickness of the membrane is determined by a digital micrometer. The overall porosity was calculated using the physical dimensions and polymer density. Addition of PEG 6000 additive could increase the porosity of the membrane, however, the addition of a large number of additives decreased the porosity of the nano-composite membrane. [29] explained that increasing the concentration of PEG 6000 made the porosity of the nano-composite membrane increase. However, when the addition of PEG 6000 was continued, the result was that the phase separation in the nano-composite membrane was delayed which caused the porosity to become low. The porosity increased due to the addition of additives that could enlarge the pores on the membrane. At high concentrations of additives, the additives will agglomerate and cause the additives to not diffuse completely so that the pore size becomes smaller.

4. CONCLUSION

Effect of Polyacrylonitrile-Fe2O3 Membrane Concentration on Membrane Performance. Flux decreases with increasing Fe2O3 concentration. The longer the filtration time, the pressure of the filtration process will also increase.

The effect of ultra violet irradiation on the performance of Polyacrylonitrile-Fe2O3 membrane for oil refinery wastewater treatment causes the membrane flux value to increase but the rejection value to decrease.

The effect of the addition of PEG on the improvement of work and membrane characterization in oil refinery wastewater treatment. Test Results Analysis of mechanical membrane The ratio of decreasing flux values on the graph is more stable from the beginning of the filtration to the end of the filtration. This phenomenon indicates that there is no accumulation on the membrane surface. A stable flux reduction ratio is important for membrane performance.

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