



# Synthesis of Polyamide- $\text{Al}_2\text{O}_3$ Nanocomposite Membranes Using the Nanofiltration Phase Separation Method

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**Abstract.** The development of Polyamide- $\text{Al}_2\text{O}_3$  nanocomposite membranes into a solution for treating oil and gas wastewater into clean water that can be flowed back into the sea or reused as a source of clean water using simple operations, without additives, cost-effective, and has high productivity. Polyamide- $\text{Al}_2\text{O}_3$  membranes have been successfully synthesized. This study aims to find out the effect of  $\text{Al}_2\text{O}_3$  concentration on the process of treating oil and gas wastewater into clean water using Polyamide nanocomposite membranes, the performance of Polyamide- $\text{Al}_2\text{O}_3$  nanocomposite membranes with the addition of the active substance PEG and PVA and characterization of Polyamide- $\text{Al}_2\text{O}_3$  nanocomposite membranes through mechanical membrane, morphology, and FTIR assays. The membrane that has been made is characterized using tensile strength, contact angle, FTIR, and SEM-EDX. The best result of polyamide membrane tensile strength is Polyamide 18% with a young modulus value of 0.16 MPa. The addition of  $\text{Al}_2\text{O}_3$  to the Polyamide membrane was able to reduce the hydrophilicity properties of the membrane from  $95.92^\circ$  to  $67.2^\circ$ , increasing the flux value by  $41.886 \text{ L/m}^2 \cdot \text{h}$ , and percent rejection by 99.8%. The SEM-EDX result of Polyamida membranes has small elongated finger-like pores and a tighter structure, while Polyamide- $\text{Al}_2\text{O}_3$  has sponge-like pores and larger pore sizes.

**Keywords:** Membranes · Polyamide ·  $\text{Al}_2\text{O}_3$  · wastewater

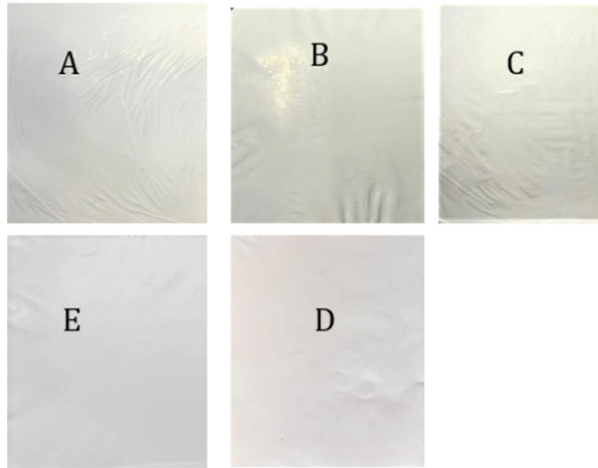
## 1 Background

The petroleum industry involves purifying water treatment with nanofiltration membranes. Membrane is an appropriate technology for water treatment. This technology uses simple operation, no additives, cost-effective, has high productivity. The advantage of this membrane is that it can be used for brackish water, wastewater, seawater desalination, etc. (Hidayah et al., 2021). Polyamide (PA) is a very popular type of polymer used as a membrane material (Zahid et al., 2018). PA has the advantages of good

mechanical properties, resistance to high temperatures up to 200°C, strong resistance to chlorine and other chemicals and easy to be moduled in configuration (Zhao et al., 2018). Hydrophilic metal oxide nanoparticles, such as Al<sub>2</sub>O<sub>3</sub>, zeolite, and TiO<sub>2</sub>, are among the attractive membrane materials because of their high water permeability. Al<sub>2</sub>O<sub>3</sub> has been reported to be a good absorbent. In addition, incorporating nanomaterials into the membrane improves permeability, thermal stability, resistance to fouling, and mechanical properties, as well as provides new functions such as self-cleaning and degradation of contaminants (Karki et al., 2019). Al<sub>2</sub>O<sub>3</sub> is widely used as a fire retardant. This is because Al<sub>2</sub>O<sub>3</sub> has various features, namely having good corrosion resistance in various environments, having excellent bioinert properties and having good high temperature resistance and good electrical resistance. In addition, Al<sub>2</sub>O<sub>3</sub> is known for two impressive properties, namely hydrophilic and covalent bonding properties. (Dai et al., 2016). Al<sub>2</sub>O<sub>3</sub>-based technologies besides those already mentioned, can also play a major role in making water purification processes economically feasible.. Currently, processing with aluminum is a promising solution, effective and reliable when compared to traditional processes in terms of cost-effectiveness, environmental preservation and the ability to remove contaminants such as carbon dioxide and hydrogen sulfide in various operating conditions (Esfahani et al., 2019). The solution to avoiding the environmental impact of this type of wastewater is first minimization, secondly reuse and finally and as a last resort, disposal. Prior treatment was required to remove the various toxic compounds present in oil and gas wastewater, although unfortunately, the law only concerns oil and gas, and not other substances at this time. However, recently more attention has been paid to the parameters and other hazardous components of oil and gas wastewater (Rahi et al., 2021). The development of the Polyamide-Al<sub>2</sub>O<sub>3</sub> nanocomposite membrane is expected to be a solution for treating oil and gas waste water into clean water which can be flowed back into the sea or reused as a source of clean water using simple operations, without additives, cost-effective, and has high productivity. Membrane technology still has many drawbacks, one of which is the occurrence of fouling. The focus of this research is to develop a polyamide-Al<sub>2</sub>O<sub>3</sub> nanocomposite membrane using a dip-coating technique. This also underlies this research to examine the use of materials that can overcome membrane fouling.

## 2 Methodology

The research was divided into three stages, namely the stage of making membranes made from polyamide-Al<sub>2</sub>O<sub>3</sub> stages of membrane characterization, and the stage of membrane application for oil and gas wastewater treatment. At the stage of making a Polyamide (PA) membrane begins with making a printed solution consisting of PA as a polymer with a PA composition of 18%wt. Addition of nano Al<sub>2</sub>O<sub>3</sub> 0.1, 0.5, 1.5%wt for PA. Poly ethylene glycol (PEG) 4000, 6000 with 5%wt for matrix membrane PA and the rest solvent n-methyl-2-pyrrolidone (NMP). Membrane printing through phase inversion. This method is done by putting a dopt solution membrane into the glass plate with a casting knife, then the glass plate is dipped in a coagulation bath. The addition of UV irradiation 1, 5, 10 min. Finally, the addition of Polyvinyl alcohol with a concentration of 1, 2.3%wt. The next stage is characterization by determining flux and



**Fig. 1.** (a) Polyamide 16%, (b) Polyamide 18% (c) Polyamide 20% (d) Polyamide 22% (e) Polyamide 24%

rejection, scanning electron microscopy (SEM), contact angle, fourier transform electron microscopy (FTIR) and mechanical tests. After that, a membrane application test for oil and gas wastewater treatment is carried out. The method of research schematically can be seen in Fig. 1. This chapter also describes the materials and equipment used, as well as the work procedures and data analysis that will be carried out.

## 2.1 Materials Used

The materials used in this study include: Polyamide (PA), n- methyl-pyrrolidone (NMP), oil refinery wastewater and polyethylene glycol (PEG) 4000 and 6000, Polyvinyl alcohol (PVA), nano  $\text{Al}_2\text{O}_3$ .

## 2.2 Tools Used

The tools used in this study are turbidimeters, magnetic stirrers, glass plates, coagulation tubs, casting knives, tape, ovens, SEM equipment, FTIR equipment, cross-flow titration equipment, UV-VIS spectrophotometer equipment, gas chromatography equipment.

## 2.3 Manufacture of Polyamide(PA)- $\text{Al}_2\text{O}_3$ Membrane Nanocomposites

The process of making PA- $\text{Al}_2\text{O}_3$  membrane nanocomposites uses the phase inversion method. The initial research was carried out by making a dope, a PA polymer material with a concentration of PA 18% wt. This research makes membranes with polymer materials by dissolving PA plus Nano  $\text{Al}_2\text{O}_3$  dissolved with NMP so that the pores are distributed. The solution was mixed until homogeneous, then the best PEG and the best PVA were added, then the solution was allowed to stand at 36 °C so that the air bubbles disappeared (debubbling). Settled within 24 h. Casting by removing the solution

is poured onto a glass plate, leveled with a casting knife, then demixing is carried out, namely precipitation of the molding solution into the coagulation bath). The demixing process is the immersion of the membrane sheet on a glass plate into a coagulation bath containing non-solvent. The membrane is soaked until it separates from the plate glass and then dried and then put in water at a temperature of approximately 180 °C for the annealing process. In order to get a stable membrane.

## 2.4 Membrane Characterization

Membranes designed for oil refinery wastewater purification are characterized in advance to know the specifications on membrane nanocomposites. These characteristics include determination of membrane flux and rejection, morphological structure of scanning electron microscopy (SEM), contact angle, fourier transform electron microscopy (FTIR) and mechanical tests.

## 3 Discussion

The manufacturing process of Polyamide- $\text{Al}_2\text{O}_3$  membrane uses the fas a inversion method to obtain a stable membrane, in addition to hydrophilicity tests. The hydrophilicity of the membrane is measured by measuring the contact angle between the surface of the membrane and water. The effect of ultraviolet (UV) irradiation on membrane performance is very beneficial because membrane fouling is a barrier in ultrafiltration (UF) applications for water treatment. Ultraviolet (UV) oxidation in this study was used as a pretreatment to control membrane fouling caused by natural organic matter in surface water. The influence of UV pretreatment on quantity and characteristics was investigated in terms of dissolved organic carbon, fluorescent spectrum, molecular weight distribution and hydrophobicity (Tian et al., 2018). Turbidity of oil refinery wastewater using a turbidimeter tool with NTU value analysis before passing through the membrane hybrid and after. In this chapter we will discuss the manufacture and characterization of the Polyamide- $\text{Al}_2\text{O}_3$ . The membrane that has been created is then characterized by tensile strength, contact angle, flux and percent rejection. FTIR to find out the functional groups on the membrane and SEM-EDX to find out the cross section and what elemental components are contained in the membrane.

### 3.1 Membrane Manufacturing

Membranes are one of the easiest alternatives to use for sewage treatment. Polyamide membrane is one of the most important materials applied to industrial waste treatment because it has strong thermal and hydrolytic stability, and has good mechanical properties. In this study, a study was carried out on the manufacture of powdered Polyamide membranes as the main material dissolved with N NMP solvent. The research was conducted at the Basic Chemistry Laboratory of UIN Walisongo Semarang. At the stage of membrane manufacturing is carried out in two stages, namely the manufacture of

membranes. Polyamide and Polyamide-Al<sub>2</sub>O<sub>3</sub>. For more detailed analysis can be seen in this explanation.

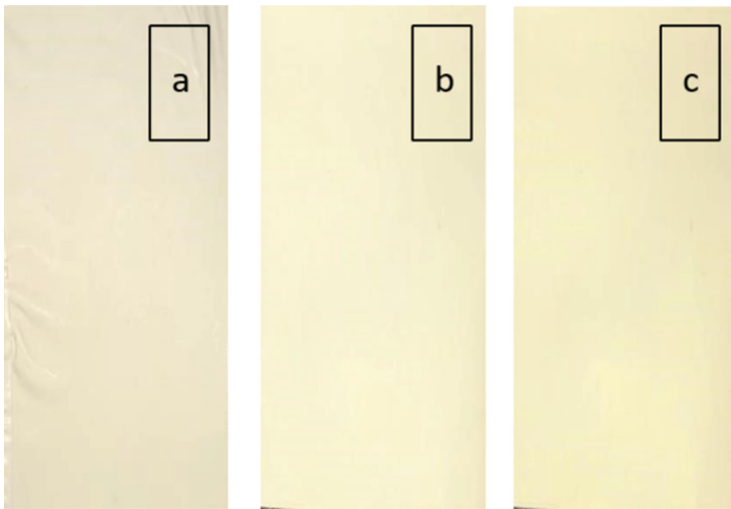
### **Polyamide-Al<sub>2</sub>O<sub>3</sub> Membrane**

In the polyamide membrane manufacturing process, the first step taken is to make a membrane with a concentration variation of 16%, 18%, 20%, 22%, and 24% with Polyamide polymer as the main material and NMP solution as solvent. Then the solution is stirred using a magnetic stirrer until the solution is homogeneous. The already homogeneous solution is allowed to stand until the bubbles on the solution disappear. Before the solution is molded, the glass plate is cleaned using acetone. This is done in order to accelerate the reaction process on the membrane. The membrane is printed using the wet phase inversion method. This method is done by molding the membrane on a glass plate and then flattening it with a stirring rod. The membrane that has been printed on the glass plate is inserted into the coagulation bath until the membrane is removed from the glass plate. After the membrane is removed from the glass plate, the membrane is removed until the membrane dries.

From Fig. 1, you can see the variation in polyamide membranes with concentrations of 16%, 18%, 20%, 22%, and 24%. The 16% polyamide membrane has a thinner texture and still looks wrinkled compared to other concentrations, this is because the higher the concentration, the thicker the membrane results on the polyamide membrane 18% is slightly thicker and the degree of wrinkles is further reduced compared to polyamide 16%. The 20% polyamide membrane produces a fairly good membrane, because the number of bubbles is decreasing, compared to the previous concentration, nmun still sees some wrinkles on the membrane. On the 22% polyamide membrane, bubbles are also decreasing and the degree of wrinkles is decreasing compared to the previous concentration. On the polyamide membrane 24% when viewed is the best membrane because the resulting membrane is thicker, the degree of wrinkles is frozen, and the bubbles begin to disappear. But to prove the membrane is best done tensile strength test. A tensile strength test on the polyamide membrane is carried out to determine the mechanical properties of the membrane. After obtaining the best polyamide membrane, the next step is to mix the polyamide membrane with silica.

After obtaining the best polyamide membrane by conducting a tensile strength test, the next step is to mix the polyamide membrane with silica. This is done because polyamides have a weakness that is hydraphobic, resulting in membrane decay and fouling easily. Therefore, testing is carried out by mixing polyamide with silica to improve performance on the membrane. The same step is also carried out for the manufacture of polyamide-Al<sub>2</sub>O<sub>3</sub>. Polyamide-Al<sub>2</sub>O<sub>3</sub> membranes are made with variations of 5%, 10%, 15% (w/w). After obtaining membranes with different variations, a tensile strength test was carried out to find out which membrane had the best mechanical properties. For polyamide-Al<sub>2</sub>O<sub>3</sub> membrane results can be seen in Fig. 2.

On the Polyamide-Al<sub>2</sub>O<sub>3</sub> membrane has a yellower color compared to the polyamide membrane. The occurrence of discoloration due to the presence of a mixture of silica nanoparticles. Of the three variations, the more concentration increases, the color produced by the membrane is also yellower and the degree of wrinkles on the polyamide-al<sub>2</sub>o<sub>3</sub> membrane is less than that of the polyamide membrane. The next step is that



**Fig. 2.** Membrane (a) Polyamide- $\text{Al}_2\text{O}_3$  5% (b) Polyamide- $\text{Al}_2\text{O}_3$  10% (c) Polyamide- $\text{Al}_2\text{O}_3$  15%

it is necessary to do a tensile strength test to find out which membrane has the best mechanical properties.

### 3.2 Membrane Characterization

In this study, characterization of the membrane was carried out which included, tensile strength test to determine the mechanical strength of the membrane, knowing the properties of hydrophobic or hydrophilic membranes with contact angle tests, knowing the functional groups contained in membranes with FTIR, knowing flux and rejection values with filtration tests, and knowing the surface of membranes and elemental components with SEM-EDX.

#### Tensile Strength Test

Tensile strength tests were performed to determine the elasticity and tensile resistance of polyamide and polyamide- $\text{Al}_2\text{O}_3$  membranes. Tensile strength tests are performed to determine the magnitude of stress and strain until they reach maximum attraction on polyamide and polyamide- $\text{Al}_2\text{O}_3$  membranes. Measurements of mechanical properties are carried out to determine the strength of the membrane. The tensile strength test aims to measure the force required to make the polyamide and polyamide- $\text{Al}_2\text{O}_3$  membranes disconnected. The tensile strength test needs to be done to find out how the strength of the membrane is against forces coming from the outside that can damage the membrane, the tighter the membrane structure, the tighter the distance between membrane molecules so as to produce a membrane that has a strong tensile strength. The nature of mechanical strength in membranes can be known by using the calculation of Young's modulus, namely by calculating the ratio of stress to strain with equation III.3. Data on the results of the tensile strength, elongation, and modulus tests on polyamide membranes can be seen in Table 1.

**Table 1.** Values of tensile strength, elongation, and *modulus of young* polyamide membranes

No	Sample name	Tensile strength (mpa)	Elongation (%)	Modulus young (mpa)
1.	Polyamide 16%	1,76	26,7	0,06
2.	Polyamide 18%	3,85	24,7	0,16
3.	Polyamide 20%	3,93	34,0	0,115
4.	Polyamide 22%	4,18	38,3	0,109
5.	Polyamide 24%	5,29	45,0	0,117

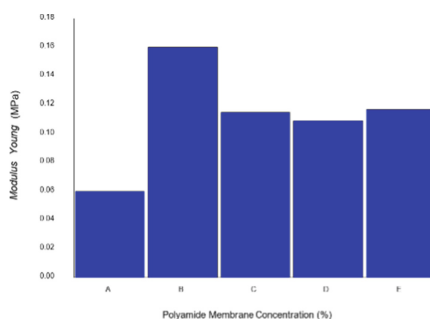
**Fig. 3.** Modulus values of young polyamide membranes at various concentrations

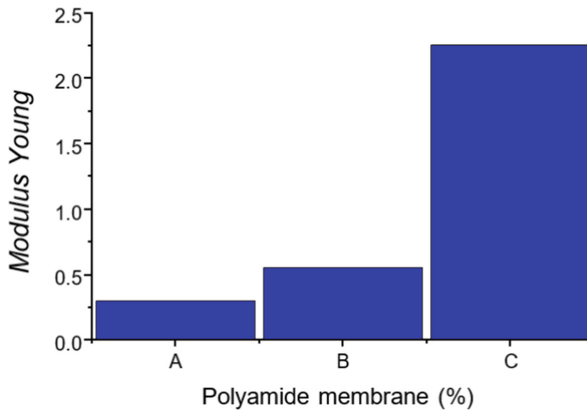
Table 1 shows that the more additions to the polyamide composition, the greater the tensile strength value. Based on the table, the result of large tensile strength is found in the polyamide membrane with a concentration of 24%. From the data obtained, the best Young modulus value is found in polyamide 18%. This is in accordance with Suryandari's research (2019) that the greater the value of the young modulus, the better the mechanical properties of the membrane. The results of the analysis of mechanical properties on the polyamide membrane can be seen in Fig. 3. Where a is 16% polyamide, b is 18% polyamide, b is 20% polyamide, d is 22% polyamide, and e is 24% polyamide.

Figure 3 shows that the modulus value of young polyamides is best found in polyamide membranes with a concentration of 18%. After obtaining the best young polyamide modulus value, polyamide- $\text{Al}_2\text{O}_3$  membranes were made with variations in the addition of  $\text{Al}_2\text{O}_3$  5%, 10%, and 15% (W/B). To obtain the best membrane results, a tensile strength test is carried out on the polyamide- $\text{Al}_2\text{O}_3$  membrane. The results of the analysis of the tensile strength of the polyamide- $\text{Al}_2\text{O}_3$  membrane can be seen in Table 2.

From Table 2 shows that the greatest tensile strength value is found in the polyamide- $\text{Al}_2\text{O}_3$  membrane with a concentration of 15%. It can be concluded that the more silica additions, the greater the tensile strength value. To determine the mechanical properties on the membrane can be expressed in Young's modulus From the data obtained, the best Young modulus value is found in the 15% polyamide- $\text{Al}_2\text{O}_3$  membrane. The results of

**Table 2.** Results of tensile strength, elongation, *young modulus* polyamide- $\text{Al}_2\text{O}_3$  membrane

No	Sample name	Tensile strength (mpa)	Elongation (%)	Modulus young (mpa)
1.	Polyamide- $\text{Al}_2\text{O}_3$ 5%	3,52	11,7	0,30
2.	Polyamide- $\text{Al}_2\text{O}_3$ 10%	2,45	4,3	0,56
3.	Polyamide- $\text{Al}_2\text{O}_3$ 15%	4,53	2.0	2,26

**Fig. 4.** Effect of  $\text{Al}_2\text{O}_3$  Variation Addition on *Young Modulus* of Polyamide Membranes

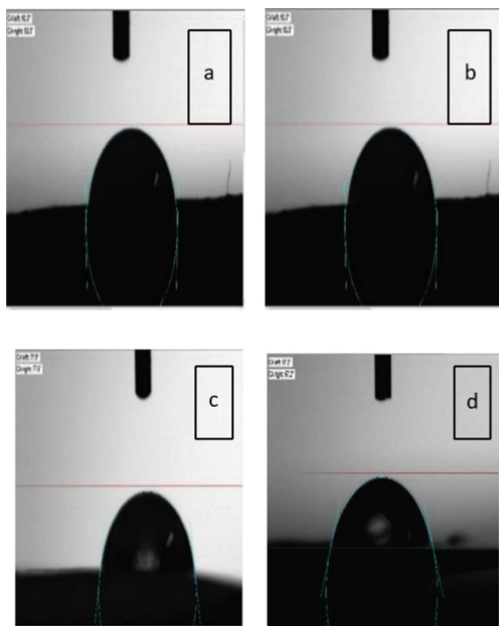
the analysis of Young's modulus values can be seen in Fig. 4 with A is polyamide-  $\text{Al}_2\text{O}_3$  5%, B is polyamide-  $\text{Al}_2\text{O}_3$  10%, and C is polyamide-  $\text{Al}_2\text{O}_3$  15%.

Figure 4 shows that the higher the concentration of addition of  $\text{Al}_2\text{O}_3$ , the greater the tensile strength value. In addition to the tensile strength value, the smaller elongation value also affects the mechanical properties of the membrane, so Young's modulus value is also getting bigger. This happens because the polymer chain has successfully bound organic compounds from silica, so the intermolecular force is even greater. This is in accordance with the theory that to determine the mechanical properties of a good membrane, namely a membrane that has a large tensile strength, and a small elongation value (Rohmah, 2021).

### Contact Angle Test Analysis

The contact angle test is one of the characterizations to determine the hydrophobic or hydrophilic properties of the membrane by measuring the contact angle formed from water when interacting on the surface of the membrane. Water contact angle testing is carried out to determine the wettability properties of the film/membrane surface. The step taken in this test is to drip voluminous water over the surface of the film/membrane. Water droplets hitting the surface of the membrane are photographed using a camera or recorded in the form of a video. This photo is then analyzed and measured the contact angle of the water. The contact angle is the angle formed between the plane of





**Fig. 5.** Figure of the contact angle of the membrane (a) polyamide, (b) polyamide- $\text{Al}_2\text{O}_3$  5%, (c) polyamide- $\text{Al}_2\text{O}_3$  10%, (d) polyamide- $\text{Al}_2\text{O}_3$  15%.

**Table 3.** Membrane contact angle test results

Membrane	Contact Angle ( $^{\circ}$ )
Polyamide	95,9
Polyamide- $\text{Al}_2\text{O}_3$ 5%	92,3
Polyamide- $\text{Al}_2\text{O}_3$ 10%	77,5
Polyamide- $\text{Al}_2\text{O}_3$ 15%	67,2

the film/membrane surface and the plane of the water droplet. From this angle can be determined the wettability properties of a membrane. Analysis of membrane wetness properties was carried out on polyamide membranes, polyamide- $\text{Al}_2\text{O}_3$  5%, polyamide- $\text{Al}_2\text{O}_3$  10%, and polyamide- $\text{Al}_2\text{O}_3$  15%. The results of the analysis of contact angles can be seen in Fig. 5.

From Fig. 5 it can be seen that the addition of silica has an effect on its hydrophilicity properties. It can be proved that the degree of each membrane is decreasing more and more. For clearer results of the analysis of the contact angles of polyamide and polyamide- $\text{Al}_2\text{O}_3$  membranes can be seen in Table 3.

Table 3 shows a decrease in the contact angle value as silica is added to the membrane. It can be concluded that the more silica additions, the larger the surface pores on the membrane, so that the resulting contact angle will be lower due to higher absorption.

**Table 4.** Results of polyamide and polyamide-al<sub>2</sub>o<sub>3</sub> membrane filtration tests

Membrane	Sample Volume (l)	Fluks Membrane (l/m <sup>2</sup> .jam)
Polyamide	0,0095	13,721
Polyamide-Al <sub>2</sub> O <sub>3</sub> 5%	0,014	20,220
Polyamide-Al <sub>2</sub> O <sub>3</sub> 10%	0,024	34,664
Polyamide-Al <sub>2</sub> O <sub>3</sub> 15%	0,029	41,886

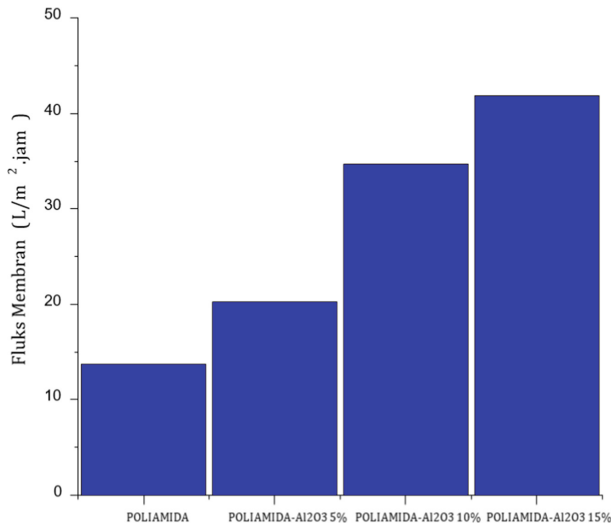
Based on Table 3, it can be seen that the research carried out according to the theory is, if the small contact angle ( $<90^\circ$ ) shows high wettability (the membrane is hydrophilic), while the large contact angle ( $>90^\circ$ ) indicates a low level of wettability (hydrophobic) (Suryandari, 2020). Based on Agustina's explanation, etc. (2019) that polyamide is one of the polymers used as the basic material for making membranes. However, polyamides are hydrophobic, causing a decrease in permeability due to the deposition of particles on the surface of the membrane which results in fouling, so that the membrane becomes easily damaged. Therefore, it is necessary to add nanoparticles to the membrane, one of which is silica which aims to improve or modify mechanical properties and improve the characteristics of the membrane which was originally hydrophobic to hydrophilic.

#### Polyamide-Al<sub>2</sub>O<sub>3</sub> Membrane Flux Test

One of the ways to determine the performance of membrane separation is by conducting a flux test on the membrane. According to Azzahra (2021), flux is a measure of the speed of a species passing through the membrane per unit area and time with the pressure gradient as the driving force. The step taken in this flux test is that the membrane is cut according to the size of the filtration cell. Then the feed solution flows into the membrane filtration at a certain pressure. Flux test was carried out by cross flow filtration for 30 min. Flux value is calculated using equation iii.5. The results of calculating the flux value can be seen in Table 4. From Table 4 the results of the filtration test with polyamide and polyamide-al<sub>2</sub>o<sub>3</sub> membranes it can be seen that the flux results on the polyamide membrane were 13.721 l/m<sup>2</sup>.h, 5% Polyamide-Al<sub>2</sub>O<sub>3</sub> membrane was 20.220 l/m<sup>2</sup>.h, 10% Polyamide-Al<sub>2</sub>O<sub>3</sub> membrane 34.664 l/m<sup>2</sup>.h and 15% polyamide-Al<sub>2</sub>O<sub>3</sub> membrane had the highest flux value with a yield of 41.886 l/m<sup>2</sup>.h.

The flux results in Table 4 can be described in the graph shown in Fig. 6.

Table 4 and Fig. 6 explain that the more silica added to the membrane, the higher the flux value. It can also be concluded that the flux value of the polyamide-al<sub>2</sub>o<sub>3</sub> membrane is higher than that of the polyamide membrane. This is because the addition of silica can increase the hydrophilicity of the membrane, so that the pores in the membrane are also getting bigger. The higher the membrane flux value, the more permeate volume that can pass through the membrane. Factors that affect higher flux values are also seen from the number and size of pores, the interaction between the membrane and the membrane solution, the viscosity of the solution and external pressure. According to Rohmah (2021), from the flux value data obtained it can be seen that polyamide



**Fig. 6.** Results of polyamide and polyamide-al<sub>2</sub>o<sub>3</sub> membrane filtration tests.

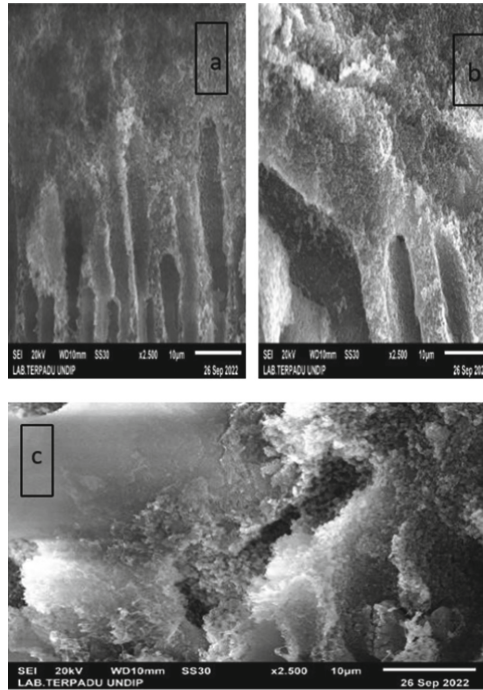
and polyamide-al<sub>2</sub>o<sub>3</sub> membranes belong to ultrafiltration membranes which have a flux range between 10–50 l/m<sup>2</sup>.h.

### Characterization of the Cross Section of the Membrane Using SEM-EDX

One way to know the structure of the membrane is to characterize the membrane using sem. In this study, analysis was carried out using a scanning electron microscope energy dispersive x-ray (sem-EDX). sem edx analysis aims to determine the pore structure, pore geometry, and surface posity found in the membrane, while the edx aims to determine the atomic components of any element present in the membrane.

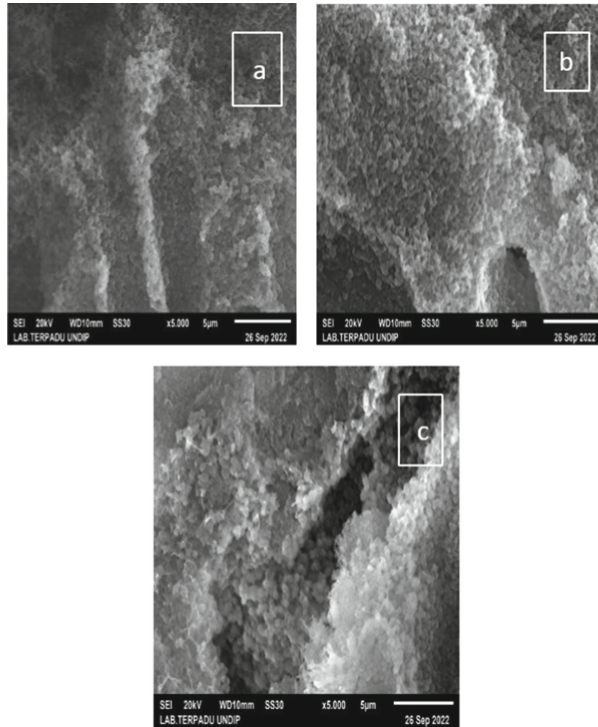
In this study, semedx analysis was performed on cross sections of Polyamide- $\text{Al}_2\text{O}_3$  membranes. SEM analysis was performed on polyamide, 5% Polyamide- $\text{Al}_2\text{O}_3$ , and 15% Polyamide- $\text{Al}_2\text{O}_3$  membranes. SEM characterization results of Polyamide- $\text{Al}_2\text{O}_3$  membranes can be seen in Fig. 7.

From Fig. 7 you can see a cross section of the membrane with a magnification of 2500x. From Fig. 7 it can be seen that each membrane, both polyamide and Polyamide- $\text{Al}_2\text{O}_3$ , is already have pores even though they have different pore sizes. There is a difference in pores in each membrane due to the addition of silica. Polyamide membranes have pores that are shaped like elongated fingers and the resulting pores are denser so that it is more difficult for water to penetrate the membrane. While the polyamide-al<sub>2</sub>o<sub>3</sub> membrane has a larger pore structure than polyamide membranes. The pore shape produced by the polyamide-al<sub>2</sub>o<sub>3</sub> membrane is also different from that polyamide membrane. Polyamide- $\text{Al}_2\text{O}_3$  membrane has pores form like a sponge. For clearer pore results on



**Fig. 7.** sem image 2500x magnification (a) polyamide, (b) Polyamide- $\text{Al}_2\text{O}_3$  5%, (c) Polyamide- $\text{Al}_2\text{O}_3$  15%

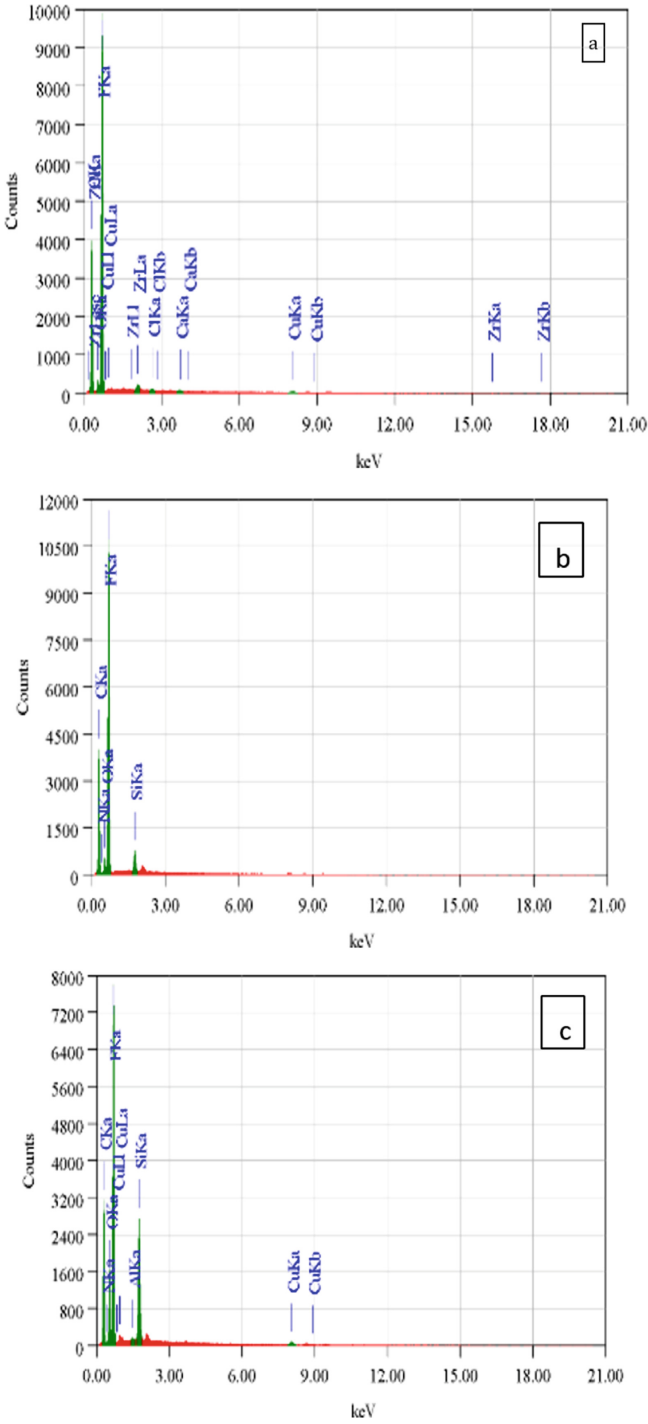
each membrane can be seen in Fig. 8. Can see a cross section of the membrane with a magnification of 5000x. From Fig. 8 it can be seen that each membrane, both Polyamide- $\text{Al}_2\text{O}_3$ , has different pores. In the polyamide membrane the pores are very tight so that in the filtration process using a polyamide membrane it is very difficult for water to penetrate the membrane. The Polyamide- $\text{Al}_2\text{O}_3$  membrane has a larger position than the polyamide membrane, but the 15% Polyamide- $\text{Al}_2\text{O}_3$  membrane has the largest pore, so that particles that pass through the membrane can be stuck in the membrane pore. This is in accordance with the research by Agustina, et al (2019) which explained that the addition of nanoparticles to the membrane had an effect on the pore structure from an elongated finger-like structure to a sponge-like structure. In Polyamide- $\text{Al}_2\text{O}_3$  membranes, the pores produced by 15% Polyamide- $\text{Al}_2\text{O}_3$  are larger than those of 5% Polyamide- $\text{Al}_2\text{O}_3$  membranes. A larger pore structure will be better for the hydrophilicity characteristics of the membrane because it has the ability to retain particles in the pore cavity and prevent water from easily penetrating the membrane pores. The addition of silica greatly affects the pores of the membrane, the more silica is added, the larger the resulting pore. This is due to the interaction between silica and polymer so as to form pores. Besides that, to determine that silica has been mixed with polyamide can be analyzed using edx. edx analysis was carried out to determine whether the components of silica elements have been mixed in the membrane. In this study, characterization was



**Fig. 8.** sem images 5000x magnification (a) Polyamide, (b) Polyamide- $\text{Al}_2\text{O}_3$  5%, (c) Polyamide- $\text{Al}_2\text{O}_3$  15%

carried out using sem-EDX to determine the elemental components of polyamide and Polyamide- $\text{Al}_2\text{O}_3$  membranes which can be seen in Fig. 9.

From Fig. 9 shows that the addition of silica has been successfully done. This can be seen in the 5% and 15% Polyamide- $\text{Al}_2\text{O}_3$  membrane edx graphs that the element Si has been detected. The graph also shows an increase in the element of Si as the concentration of silica is added to the membrane. For the results of the analysis of the components of the elements contained in the membrane more details can be seen in the table. In this chapter, we will discuss the conclusions from the research that has been carried out and suggestions that can be used as a reference for the follow-up of this research.



**Fig. 9.** Membrane edx graphs (a) polyamide, (b) Polyamide-Al<sub>2</sub>O<sub>3</sub> 5%, (c) Polyamide-Al<sub>2</sub>O<sub>3</sub> 15%

## 4 Conclusion

From the research that has been done, it can be concluded that:

1. The results of the ftir characterization of Polyamide-Al<sub>2</sub>O<sub>3</sub> membranes show the presence of si-oh peaks at wave numbers 3421.6417 cm<sup>-1</sup>, si-o-si at wave numbers 1062.6865 cm<sup>-1</sup>, sio at wave numbers 923.13432 cm<sup>-1</sup> and 497.8441 cm<sup>-1</sup>. From the sem-edx data shows that the polyamide membrane has elongated small finger-like pores and a denser structure, while Polyamide-Al<sub>2</sub>O<sub>3</sub> has pores like a sponge and a larger pore size.
2. Polyamide-Al<sub>2</sub>O<sub>3</sub> membranes have been successfully synthesized. The addition of silica to the polyamide membrane was able to increase the hydrophilicity properties, increase the flux value by 41.886 l/m<sup>2</sup>.h, and the rejection percent by 99.8% on the membrane.
3. Polyamide membrane with the addition of silica is able to filter pb2 + metal and reduce the yield of lead content with a yield of 0.09 mg/l and 0.02 mg/l which are in accordance with the requirements that can be disposed of into the environment is 0.1 mg/l.

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