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# Sustainable Acid Mine Drainage Water Reclamation Using Silica-pectin Multichannel Tubular Membrane: A Comparison of Ultrafiltration Vs Pervaporation

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### Abstract

The practice of coal mining has been demonstrated to exert a detrimental impact on the surrounding environment, particularly through the formation of acid mine drainage (AMD) ponds, which have the potential to pollute water sources. The reclamation of AMD is necessary to treat wastewater to ensure its safety for discharge into the environment and subsequent use as clean water. This study aims to treat AMD by comparing ultrafiltration (UF) and pervaporation (PV) processes utilizing silica-pectin multichannel membranes. The membranes were fabricated by coating silica-pectin sol on an inner surface of multichannel tubular support. The UF process was conducted under various pressures (1-3 bar), while the PV process was tested at various feed temperatures. Both permeate were collected and analyzed using several parameters (pH, Mn, and conductivity). The results showed that the UF process is more effective in collecting permeate flux over 136.6 L.h<sup>-1</sup>.m<sup>2</sup> at 3 bar pressure. Meanwhile, PV performs high permeate quality with Mn and conductivity rejection of 99.9 and 96.5%, respectively. Both UF and PV processes exhibit slightly increasing permeate pH with a range of 4.5-5.6. It concluded that multichannel silica-pectin membranes successfully reclamation AMD to enhance water quality. In addition, the UF process is more affordable for recycling AMD with high permeate flux, pretty good Mn, and conductivity rejection of over 95%.



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## 1. Introduction

Acid mine drainage (AMD) is regarded as a significant water contaminant in numerous locations where mining activities have occurred, historically or in the present [1, 2]. AMD's generation, release, mobility, and attenuation

are complex processes governed by a combination of physical, chemical, and biological factors [3, 4]. In general, AMD is produced by the oxidative dissolution of sulfide minerals [5]. After generation, it is very difficult, expensive, and time-consuming to suspend and/or control AMD-based contamination of surface and

**Table 1.** Initial characteristics of AMD.

Parameter	Initial Characterization	Units	Wastewater quality standards
			(Regulation Minister of Environment of Republic Indonesia No. 5 Year 2014)
pH	3.30	-	6-9
Mn	9.46	mg/L	5
Conductivity	1270	$\mu\text{S}/\text{cm}$	-

groundwater, particularly for abandoned mined land [6]. The low levels of pH and high concentration of metals and metalloids (copper, gadolinium, lithium, etc.) in mining pits with standing water lead to changing the balance of surrounding organisms and ecosystems [7].

There are several ways to treat AMD, such as the adsorption process, ion exchange, chemical precipitation process, process of electrochemical, phytoremediation process, and membrane filtration. The benefits of the membrane filtration technique, which include operation at room temperature, low energy consumption, simple operation processes, high efficiency, and small investment [8–12], have invited more consideration than other treatment techniques used to purify AMD [13]. Several process methods using membrane technology are quite commonly used in the water treatment process, including ultrafiltration and pervaporation processes. The ultrafiltration process separates components or substances using membrane technology based on the size of molecules and membrane pores influenced by pressure differences, where the components filtered during the process are a function of the size and structure of the dissolved components [14]. At the same time, pervaporation is a method of desalination of solutions using the principle of phase change to separate between water molecules and salt molecules in a vacuum atmosphere involving semi-permeable membranes selective to water and salt [15–19].

This study uses multichannel silica-pectin membranes because of several performance advantages. Multichannel membranes have more than one channel, which will increase the surface area of the membrane and result in higher flux than single-channel membranes [20–22]. This study also uses a silica-pectin thin film, in which silica has hydrophilic properties towards the water; it is necessary to add organic materials such as pectin to increase membrane stability [23–27]. Silica and organic materials such as pectin have been reported to have good hydrostability [28–30]. Pectin substances are polymeric compounds that bind water, form gels, or thicken liquids in membranes. One of several types of pectin is banana pectin, which is a natural carbon [26, 31].

Besides being easily available, pectin from banana peel waste is also more environmentally friendly than synthetic carbon and has stabilizing, thickening, and gelling capabilities [32].

This multichannel membrane's performance will be compared using ultrafiltration and pervaporation processes. Both processes have contrasting advantages and disadvantages. The ultrafiltration process tends to produce higher flux values, while the pervaporation process tends to produce lower fluxes with better filtration of substances. This study aims to compare the performance of ultrafiltration and pervaporation processes for AMD reclamation using silica-pectin multichannel membranes in reducing pH, conductivity, and Mn values.

## 2. Materials and Methods

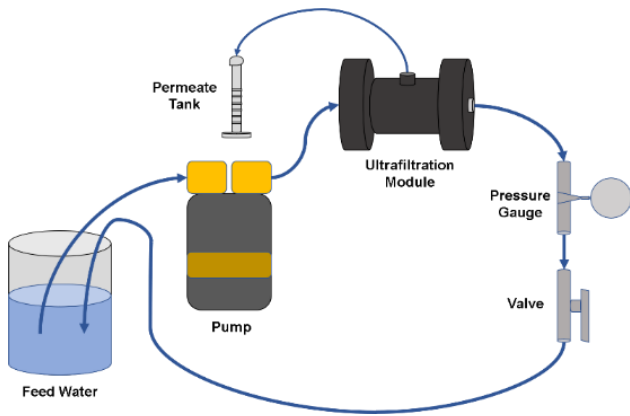
### 2.1. Chemicals and Materials

Chemicals and materials were used for silica-pectin membrane fabrication, such as tetraethyl orthosilicate (TEOS) 99% (Sigma-Aldrich), pectin from apple (Sigma-Aldrich), ethanol 97%, aqua dest, nitric acid ( $\text{HNO}_3$ ) 0.0008 M (Merck) diluted aqua dest, ammonia ( $\text{NH}_3$ ) 0.0003 M (Merck) diluted ethanol, glycerol 85% (Merck), tubular membrane support  $\text{Al}_2\text{O}_3$  (Ceramic Oxide Fabricators, Australia), with a length of 50 mm and outside diameter of 8 mm, AMD as the feed solution, and liquid nitrogen.

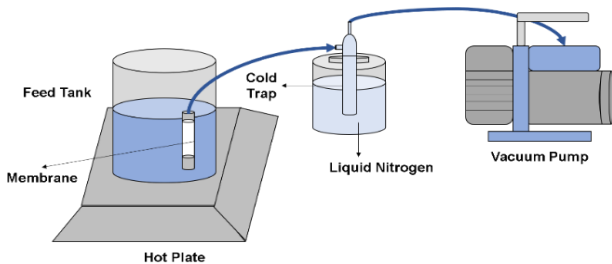
### 2.2. AMD Characterization

AMD was used as feed water in a coal mining pond in South Kalimantan, Indonesia. The parameters tested followed the quality standards of the Minister of Environment Decree No. 113 of 2003 concerning Quality Standards for AMD.

Table 1 summarizes the characteristics of AMD that was used as feed water in this work. The initial pH of AMD is extremely low, about 3.30, which does not meet the standard of clean water quality according to regulation of Minister of Environment, Indonesia No 5/2014. In addition, the Mn parameter exceeds the quality standard, which is up to 9.5 mg/L. Therefore, AMD water is required to be treated in order not to pollute the environment.



**Figure 1.** Illustration of ultrafiltration set-up.



**Figure 2.** Schematic of pervaporation equipment.

### 2.3. Preparation of Silica-Pectin Sol

The silica-pectin sol synthesis procedure was detailed in our previous work [33]; in the first step, TEOS was dropped into the ethanol whilst stirring the solution for 5 min at a temperature of 0 °C. HNO<sub>3</sub> then added to the solution and refluxed for 1 h at 50 °C. This process was followed by adding NH<sub>3</sub>, and the solution was stirred for another 2 h under the same conditions. The pH of the final sols was checked (pH 6). Pectin (0.1 wt%) was dissolved into 5 ml of glycerol whilst stirring at 360 rpm for 45 minutes at 40 °C. This mixture was added to the pure silica sol and stirred at 360 rpm for 45 minutes at 0 °C. The sol was dried in an oven at 60 °C to produce xerogels. After that, the xerogels were calcined in air at 300 °C. The initial molar ratios of silica-pectin-TEOS: EtOH : HNO<sub>3</sub>: H<sub>2</sub>O : NH<sub>3</sub>: pectin were calculated to be 1:38: 0.0008: 5 : 0.0003: 0.000026.

### 2.4. Fabrication and Characterization of Silica-pectin Multichannel Inner-coating Membrane

Silica-pectin sol was coated into the inner surface of the multichannel tubular alumina support for 2 minutes of immersion time. Afterward, the membrane was calcined at 300 °C for an hour using a vacuum furnace's RTP (rapid thermal processing) method. This process was repeated up to 4 times to obtain four layers. The membranes were characterized using FTIR spectrophotometry analysis, and the morphology structure was determined using a scanning electron microscope (SEM).

### 2.5. Ultrafiltration Performance of Silica-pectin Multichannel Membrane for AMD Reclamation

The performance of silica-pectin multichannel membranes was tested for AMD water reclamation via UF and PV processes. The multichannel silica-pectin membrane was placed into the tubular UF module and tested under a cross-flow system at room temperature. Figure 1 shows a schematic of the ultrafiltration set-up, which consists of the feed water tank, pump, permeate tank and measurer, ultrafiltration module, and valve. Using water hoses, the UF module was connected to the pump and feed water tank. AMD as feed water was placed into a beaker glass and connected to the pump and UF module. The UF pressure was adjusted with a varied range of 1-3 bar. The permeate was collected and measured every 5 minutes for 30 minutes of operation time. The permeate was measured for parameter pH.

### 2.6. Pervaporation Performance of Silica-pectin Multichannel Membrane for AMD Reclamation

The performance of silica-pectin multichannel membrane for AMD reclamation was conducted using pervaporation set-up as illustrated in Figure 2. The operation membrane system was set to dead-end. The silica-pectin multichannel membrane was immersed into a glass beaker containing AMD as feed water. A hot plate heated the feed water at 25-60 °C temperatures. The multichannel membrane was connected to a hose from a vacuum pump with the permeate flow accommodated in a cold trap immersed in a container containing liquid nitrogen. The pervaporation process was carried out for 20 minutes with a repetition of 3 times, and then the cold trap containing the permeate was collected and measured for several parameters, i.e., pH, conductivity, and Mn.

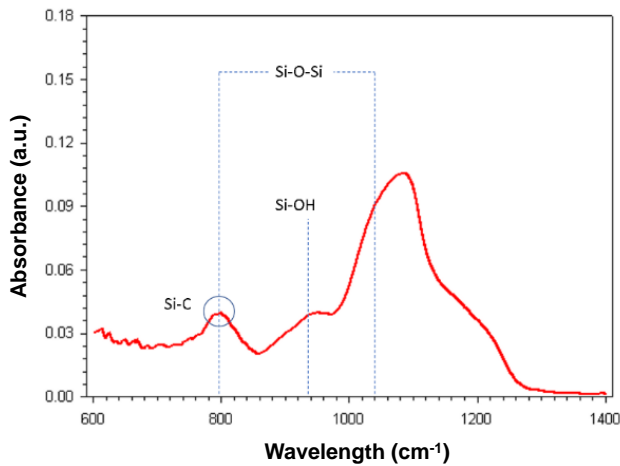
### 2.7. Flux and Rejection Calculation

The permeate flux (F) calculation is based on the permeate water flow rate in the filtration process using Equation 1. F is Flux (L.h<sup>-1</sup>.m<sup>-2</sup>), V is the permeate volume (L), A is the membrane surface area (m<sup>2</sup>), and t is Time (hour).

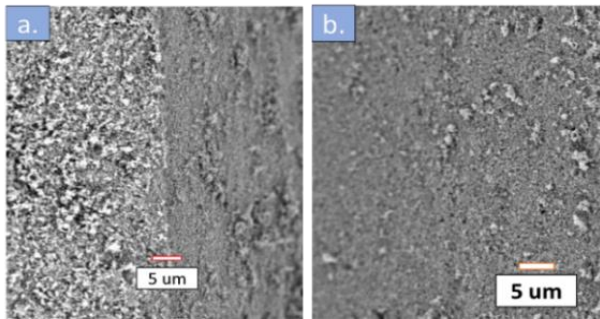
$$F = \frac{V}{(A \times t)} \quad (1)$$

Rejection of contaminant is measured using Equation 2, in which R is rejection (%), C<sub>f</sub> and C<sub>p</sub> stand for feed concentration and permeate concentration (mg/L), respectively.

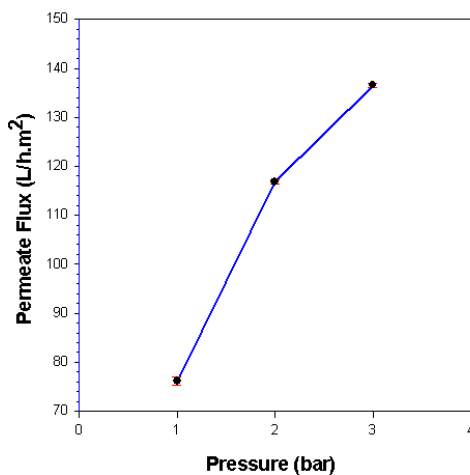
$$R(\%) = \frac{(C_f - C_p)}{C_f} \times 100 \quad (2)$$



**Figure 3.** FTIR spectra of silica-pectin multichannel membrane.



**Figure 4.** Membrane morphology using SEM analysis: a) cross section b) surface area.



**Figure 5.** Permeate flux of AMD reclamation by ultrafiltration process under various operation pressure.

### 3. Results and Discussion

#### 3.1. Characteristic and Morphology Structure of Multichannel Silica-Pectin Membranes

The chemical functional group silica-pectin multichannel membranes were characterized using FTIR spectra with a wavelength range of 600-1400  $\text{cm}^{-1}$ . FTIR analysis characterizes the functional groups or chemical groups of the silica-pectin membrane. The graph of FTIR test results

can be seen in [Figure 3](#). It can be seen that the spectra length starts from 600-1400  $\text{cm}^{-1}$ . The graph shows the chemical structure of siloxane (Si-O-Si) at wavelengths 1040  $\text{cm}^{-1}$  and 796  $\text{cm}^{-1}$ . Then, the silanol structure (Si-OH) is found at a wavelength of 930  $\text{cm}^{-1}$ . Then there is a carbon structure at a wavelength of 796  $\text{cm}^{-1}$ .

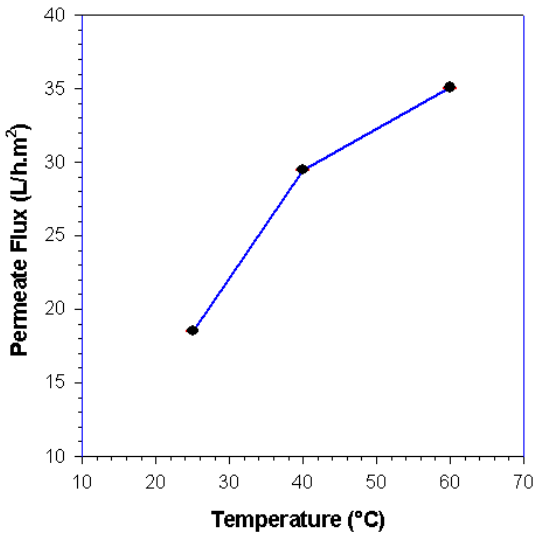
Silanol and siloxane are related to the pore size of the resulting membrane. The presence of silanol and siloxane content in the membrane has the ability to regulate membrane pores to be larger or smaller. The silanol content in the membrane tends to produce smaller pore sizes, also known as micropores. Meanwhile, siloxane forms will produce larger pores. Carbon plays a role in strengthening the silica matrix on the membrane [34].

In [Figure 4a](#), the morphology of the multichannel membrane with a scale of 5  $\mu\text{m}$  cross-section does not show any damage or cracks in the membrane. There is also a difference in pore size on the outer and inner surfaces of the membrane. In [Figure 4b](#), the multichannel membrane with a scale of 5  $\mu\text{m}$  in the surface area section where the surface looks rather smooth is caused by the inner-coating process. Previous research conducted by Akhbar [35] used a silica-pectin membrane with a dip coating method, while in this study, we used the inner-coating method.

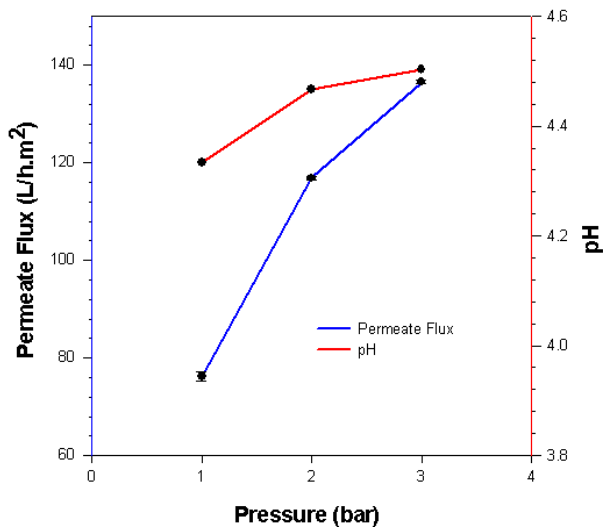
#### 3.2. Comparison performance of Ultrafiltration Vs Pervaporation for AMD Water Reclamation Using Silica-pectin Multichannel Membranes

This section evaluated the performance of both the UF and PV processes for AMD water reclamation using a silica-pectin multichannel membrane. The performance of the membrane process was measured through permeate flux and contaminant rejection. The permeate flux of the UF process for AMD reclamation at various operation pressures is shown in [Figure 5](#). As the operation pressure of UF (1-3 bar) increases, the permeate flux of AMD also increases. These are the flux results of the ultrafiltration and pervaporation process of the silica-pectin multichannel membrane.

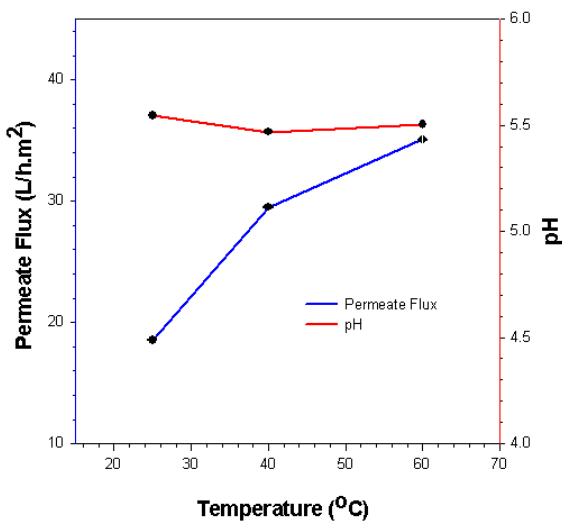
[Figure 5](#) shows the condition where there is an increase in permeate flux value caused by the greater the operating pressure, the greater the thrust force of water against the membrane. The greater the thrust force and large membrane surface area, the greater the permeate flux also increases proportionally. This condition of increasing permeate flux will reach a saturation point, which is a condition where the membrane will reach the limit of filtering ability due to the condition of the pores on the membrane surface that have fully bonded with the deposited pollutant particles. The increase in the



**Figure 6.** Permeate flux of AMD reclamation by pervaporation process under various feed temperature.



**Figure 7.** Permeate evaluation of UF through flux and pH at various pressure operation.



**Figure 8.** Permeate evaluation of PV through flux and pH at various pressure operation.

permeate flux value as the pressure increases is caused by the increasing value of energy that can be produced and given to the waste stream so that the permeate can be pushed more strongly through the membrane pores [36].

In addition, adding pressure over a long period can increase the chances of membrane fouling. Fouling occurs due to the clogging of membrane pores during the process of separating substances, which reduces the membrane's ability to filter particles. Particles that accumulate on the surface of the membrane will increasingly thicken, preventing the feed water from passing through the membrane and reducing the membrane flux value [37].

The increase in operational temperature leads to a corresponding rise in water flux, as illustrated in Figure 6. This is in accordance with research [38] where it is mentioned that the greater the driving force of water accompanied by increased vapor pressure, the water passing through the water surface is also greater so that the evaporation rate of water also increases dramatically, which will also later convert the water vapor back into water or permeate. However, a very high feed water temperature can also reduce the effectiveness of the membrane because it will decrease the membrane's durability so that it is more quickly damaged and not durable. The increase in permeate flux will also not be too significant because a maximum water limit can pass through the membrane surface as particle buildup occurs on the membrane.

The ultrafiltration process using silica-pectin membranes can increase the pH value of the AMD treatment process, as shown in Figure 7. However, this increase is not significant, given that the initial pH of AMD is 3.3. In the operating pressure of 1-3 bar, the increase in pH value is very small, so the operating pressure does not affect the increase in pH value. Low pH can also be caused by the presence of micromolecular content in the water [39], where the micromolecular content that may be acidic can only be partially filtered by the membrane. The pH value of the test results through the ultrafiltration process is also quite far below the quality standard of Minister of Environment Decree No. 113 of 2003, with a standard value of 6-9. So, in the ultrafiltration process, it is necessary to pre-treat the acid mine water to be processed so that the pH value produced in the ultrafiltration process can meet the quality standard.

Figure 8 shows that the pervaporation process using a silica-pectin membrane can significantly increase the AMD treatment process's pH significantly from the initial pH of AMD, which is 3.3. There is also no significant

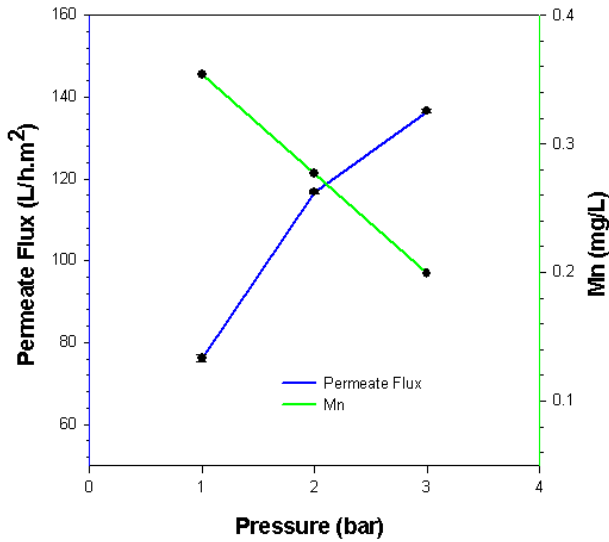


Figure 9. Permeate evaluation of UF through flux and Mn concentration at various pressure operation.

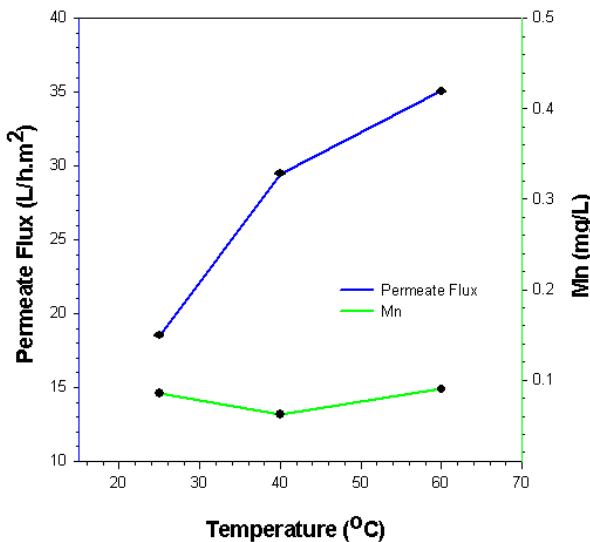


Figure 10. Permeate evaluation of PV through flux and Mn concentration at various pressure operation.

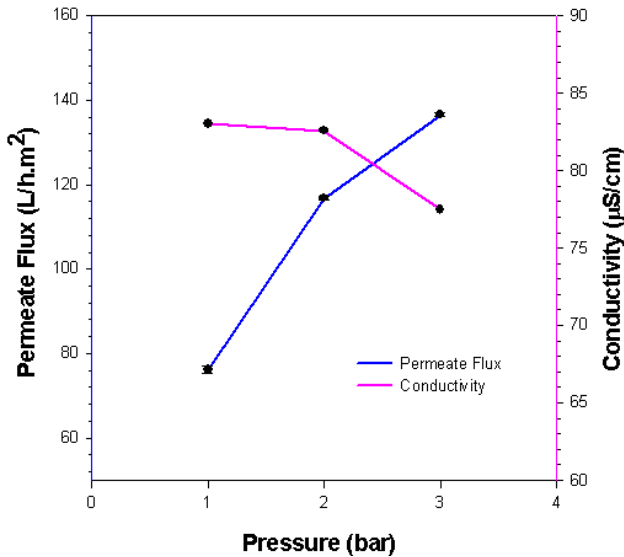
difference in the temperature range of 25 °C, 40 °C, and 60 °C, so it can be concluded that the pervaporation process in the temperature range does not really affect the decrease in pH value. The pH value of the test results through the pervaporation process is also almost close to the quality standard value of Minister of Environment Decree No. 113 of 2003, with a standard value of 6-9. The increase in pH after similar things cause the pervaporation process in the ultrafiltration process. Also, an increase in temperature decreases the solubility of CO<sub>2</sub> in water [40]. Where CO<sub>2</sub> also plays a role in the higher pH of water. Thus, the increase in pH value is greater than the result of the ultrafiltration process. Although there is an increase in pH value, this value does not meet the quality standards.

The ultrafiltration process utilizing silica-pectin membranes significantly reduces the Mn concentration in the AMD treatment process results, as illustrated in Figure 9. Starting from an initial value of 9.46, the reduction achieves an impressive 98%. The range of values of the decrease in the value of Mn in 1-3 bar operating pressure is not much different so the operating pressure does not affect the decrease in the value of Mn. The Mn value from the test results through the ultrafiltration process has also met the quality standard value of Minister of Environment Decree No. 113 of 2003 with a standard value of 4 mg/L. In general, the size of Mn metal particles in water is about 0.05-0.4 μm [41], which silica-pectin membrane pore is 0.008-0.01 μm [20], and the possibility of the heavy metal content is oxidized and will form compounds with a larger size [39] and so most of them will be filtered by the membrane.

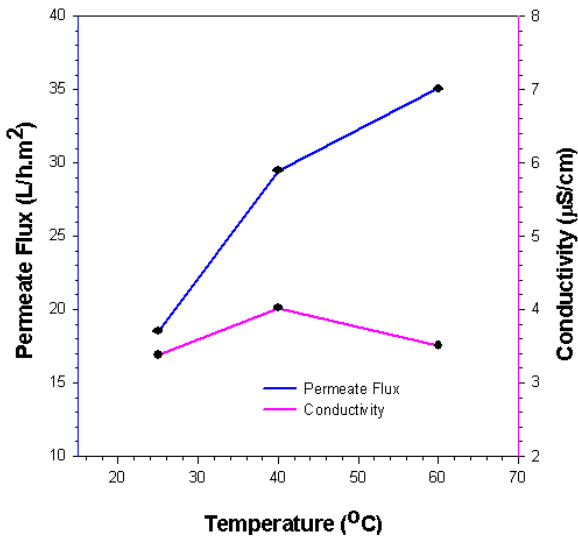
Figure 10 shows that the pervaporation process using silica-pectin membranes can reduce the value of Mn in the results of the AMD treatment process very significantly from the initial AMD value, which is 9.46, and the reduction value reaches 99%. Although the value of Mn reduction at temperatures of 25 °C, 40 °C, and 60 °C is relatively the same in that temperature range, it does not affect the decrease in Mn value. The Mn value from the test results through the pervaporation process has also met the quality standard value of Minister of Environment Decree No. 113 of 2003 with a standard value of 4 mg/L. The reduction of Mn in the pervaporation process is similar to that happened in the ultrafiltration process where Mn particles were bigger than the membrane pore, which in general Mn particles is about 0.05-0.4 μm [41], and silica-pectin membrane pore is 0.008-0.01 μm [20]. This is also supported by the phase change of water from liquid to vapor, so it filters a bit more Mn particles during the pervaporation process.

High manganese (Mn) content in water can cause or have a negative impact on health; for example, manganese substances can cause liver problems and can even cause liver cancer, as well as irritation to the eyes and skin. High Mn content in water can occur in poor environmental conditions. If feces pollute groundwater and surface water used by the community, bad bacteria will automatically spread to water sources used for household purposes. In the long run, poor-quality water can cause diseases such as bone loss, tooth corrosion, anemia, and kidney damage. This is due to toxic heavy metals beyond what the body can tolerate and the deposition of these metals in the kidneys [42].

Figure 11 shows that in the ultrafiltration process, the lowest conductivity value of water occurs at a pressure of 3 bar with a conductivity value of 77.24 μS/cm. The



**Figure 11.** Results of conductivity value in ultrafiltration process.



**Figure 12.** Results of conductivity value in the pervaporation process.

highest conductivity value occurs at a pressure of 1 bar with a conductivity value of 83.04 µS/cm. It is also known that there is a decrease in the conductivity value of each operating pressure where for 1 bar pressure to 2 bar pressure, there is a decrease in conductivity value of 0.01%, 2 bar pressure to 3 bar pressures, there is a decrease of 0.06%, and 1 bar pressure to 3 bar pressure there is a decrease of 0.07%. Overall, the ultrafiltration process can reduce the conductivity value from the initial conductivity value of 1270 µS/cm by 83% for 1 bar pressure, 82% for 2 bar pressures, and 77% for 3 bar pressures.

As shown in Figure 12, the pervaporation process resulted in the lowest conductivity value located at 25 °C with a conductivity value of 3.38 µS/cm. The highest conductivity value is located at 40 °C with a 4.02 µS/cm

conductivity value. In addition, it can also be seen in the figure that the conductivity value experiences very little increase and decrease for each feed water temperature, where for 25 °C to 40 °C there is an increase in conductivity value of 0.19%, 40 °C to 60 °C there is a decrease of 0.13%, and 25 °C to 60 °C there is an increase of 0.04%. Overall, the pervaporation process can reduce the conductivity value from the initial conductivity value of 1270 µS/cm by 99% for all three temperature variations. This value is comparable to research conducted by Akhbar (2020), in which removal efficiency also reached 99.9%.

Conductivity measurement in water aims to measure the ability of ions in water to conduct electricity and can predict the mineral content contained in water. The higher the conductivity value in water, the more dangerous it will be because high mineral and metal content can precipitate and damage the kidneys. According to WHO, the conductivity threshold value for drinking water sources is 1500 µS/cm. Electrolyte content consists of mineral salts dissolved in water; this content is related to the ability of water to conduct electric current. The more mineral salts dissolved in water, the higher the conductivity of the water. A high conductivity value causes water to easily conduct electricity and indicates a high salt content. High salt content in water will also cause the water to taste salty, so it is unsuitable for consumption. In addition, the impact of high-water conductivity values will cause a decrease in animal diversity in water bodies.

**4. Conclusions**

The ultrafiltration process is affordable for treating AMD by removing Mn and conductivity. In addition, the UF process offers a high permeate flux of about 4-fold times that of the pervaporation process. However, the pH parameter still cannot meet the quality standards for both processes. The ultrafiltration process is more recommended in AMD treatment due to more economical and efficiency requirements. This result also concluded the AMD reclamation is successfully treated via both membrane processes, either UF or PV, with significant Mn and conductivity degradation over 93%.

**Author Contributions:** Conceptualization, M.M. and A.R.; methodology, D.E.Z.Z. and M.E.; formal analysis, D.E.Z.Z.; investigation, D.E.Z.Z., A.D.A., U.K. and R.S.K.P.X.X.; data curation, M.E.; writing—original draft preparation, D.E.Z.Z.; writing—review and editing, M.E. and A.R.; visualization, F.R.M.; supervision, M.E. and M.M.; funding acquisition, M.E. All authors have read and agreed to the published version of the manuscript.

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