Chemical cleaning to evaluate the performance of silica-pectin membrane on acid mine drainage desalination

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Abstract. Pervaporation process is an excellent and potential way applied for desalting acid mine drainage water. Nevertheless, the water flux was reduced gradually due to the issue of membrane fouling. To resolve this problem, cleaning process was chosen to maintain the water flux of silica-pectin membranes. This study aims to recover the water flux and salt rejection of the silica-pectin membranes via chemical cleaning process applied for acid mine drainage water desalination with various temperature of feed water (25-60 °C). Silica-pectin membrane was formulated by employing TEOS functioning as silica precursor and pectin as carbon template from banana peels. Chemical cleaning of the membrane carried out by employing TiO\textsubscript{2} solution + UV light radiation for an hour. Performance of the silica-pectin membrane was evaluated via pervaporation process under dead-end system. The performance of silica-pectin banana peels membrane found flux recovery from 10.6 kg.m^{-2}.h^{-1} and flux recovery of 17.54 kg.m^{-2}.h^{-1}. It shows that flux recovery higher than before backwashing process. Also, silica-pectin membrane results in all of the salt rejection <99 %. It is concluded that the chemical backwashing process is important to apply to recover the water flux of membrane, also, this process considers to save and reduce the operational costs.

1. Introduction  
South Kalimantan, Indonesia has potential of natural mineral resources, it is found that the mining activities is still more important for economy region. Generally, mining activities in South Kalimantan, Indonesia using open pit mining method and generated Acid Mine Drainage (AMD) [1]. The AMD has negative effect to the environmental [2]. The characteristics of AMD on production activities/pit area have pH $\pm$ 4.28, high content of Fe reaching 9.1 mg/L and Mn content of 6.4 mg/L [3]. Modernise, product AMD has been processed using membrane technology with desalination process to reduce fouling or pollutants AMD [4]. Pervaporation in membrane desalination has preferred technique to convert to be portable water [5–9]. The
pervaporation process has advantage of the difference partial pressure from the pump as the driving force in the feed water and the permeate [5, 9–11]. Silica membrane with Tetraethyl orthosilicate (TEOS) as main precursor in the polymerisation process have a longer life time for desalination water [12]. Nonetheless, silica still has limitation due to low hydro-stability properties [13], consequently a new innovation by addiction of pectin into the silica membrane can increasing hydro-stability properties of membrane [14, 15]. The carbon content in pectin includes neutral sugar chains derived from complex polysaccharide compounds [16] strengthen the pore structure of the membrane and makes the membrane pore resistance stronger and has high salt rejection [4, 17, 18]. Hence, the large of pollutants will decrease water flux and performance of membrane, resulting in membrane fouling [19, 20].

Generally, fouling membrane type are reversible and irreversible. Particulates are deposited on the membrane such as colloids, macromolecules, salts and microorganisms. In other research there are various types of particulates that have an impact on membrane surface fouling, including inorganic particulates, dissolved organic matter, and dissolved solids [21, 22]. Impurity particles on the membrane surface impact on decreasing the permeate flux of the membrane, the difficulty of operating performance and the short life time and make decrease productivity of the system [23, 24]. Fouling membrane may reduce using backwashing membrane or chemical cleaning membrane [25]. There are type of backwashing membrane process, such as: a physical backwashing and chemical cleaning, but pollutants stronger adhering to the membrane surface, needed chemical materials to optimally cleaning. Especially photocatalytic and catalyst titanium dioxide (TiO₂) is a material that has advantages to removed fouling membrane [26]. The other advantages of using photocatalytic and TiO₂ such as catalyst in membrane separation can be recycled for reuse and increase flux recovery in the membrane [27].

Photocatalytic is a method that had founded with various applications in the reduction of persistent organic micro pollutants [28]. Concept of photocatalytic + TiO₂ as catalyst utilizes the concept of generating energy bandgaps in the excitation of photons which have semiconductor materials [29]. Photocatalytic-membrane application rehabilitated performance of the membrane [30]. In addition, TiO₂ as well as environmentally friendly catalyst [31, 32], and TiO₂ catalyst can reduce the activation energy which accelerates the reaction and pollutants are oxidised to carbon dioxide and water [33]. Utilising photocatalytic + catalyst TiO₂ methods in reducing pollutants, this work is aimed at water flux and salt rejection recovery of silica-pectin membrane by applying chemical backwash process evaluated on acid mine drainage water desalination with various feed water (25 - 60 °C).

2. Methodology

2.1. Chemicals and materials

Acid Mine Drainage (AMD) taken from South of Kalimantan-Indonesia specially at the Kintap region. Membrane support in this study from a macro-porous alumina tubular with pore size<100 nm. Membrane. Silica-pectin sols is fabricated from Tetraethyl Orthosilicate (TEOS, 99 %, Sigma-Aldrich), ethanol (EtOH, 96 %), ammonia (NH₃, 0.0003 M, Merck) and citric acid (HNO₃, 0.0078 M, Merck), pectin from banana peels (concentration 0.1 % and 0.5 %), glycerol (85 %, Merck). Chemical cleaning membrane in this study using liquid titanium dioxide (TiO₂, Merck) with photocatalytic UV (type C Philip 18 Watt).

2.2. Fabrication and characterisation silica-pectin membrane

Sol-gel membrane silica-pectin was method to product thin film membrane. pH sols at final process adjusted to pH ± 6. In other side, preparing carbon template from pectin which extract from banana peels. Prepared glycerol at 50 °C for 90 times to mix pectin-glycerol (0.1 % and 0.5 % pectin concentration). Final molar ratio was conducted from our previous work which become EtOH : TEOS :H₂O :HNO₃ :NH₃ : pectin, measure 38: 1: 5: 0.0008: 0.0003: x, where x was variation of pectin from banana peels concentration with
0.0063 grams to concentration 0.1 % and 0.032 grams to concentration 0.5 % [4]. Finishing process, the silica-pectin sols was coating in tubular membrane 1 hour for 1 layer until 4 layers. Membrane was characterisation preparing with dried sols into oven for 24 hours to product xerogel. Xerogel was calcined at 300 °C and 400 °C temperature using Rapid Thermal Processing (RTP). Finally, xerogel was ready to be characterised using FTIR spectra.

2.3. Membrane pervaporation
Performance and evaluation of membrane silica-pectin determine using pervaporation process. AMD water is used to determine the resistance and ability of silica-pectin membrane with measure of water flux, water flux recovery, and salt rejection. Feed water temperature was variations were carried out at room temperature (25 °C), 40 °C and 60 °C. Pervaporation was carried out for 20 minutes. Set up pervaporation was design as shown in figure 1.

\[ F = \left( \frac{M}{A dt} \right) \]

Wherein, mass of flux permeate (kg) collected in the cold trap (M), A is an activated-area on surface silica-pectin membrane (m²) and operational times was symbol of dt (h). Salt rejection in silica-pectin banana peels membrane measured with the equations (2):

\[ R = \left( \frac{CF - CP}{CF} \right) \times 100 \% \]

Wherein, \( CF \) is a permeate solution and \( CP \) is a retentate solution. Salinity of solution can measure using conductivity meter (OHAUS SF300C-G)

2.4. Chemical cleaning (Backwashing membrane)
Chemical cleaning to recovery performance of membrane silica-pectin prepared with hired submersion of membrane silica-pectin using titanium dioxide solid and accompanied by irradiation photocatalytic UV for an hour. Set up of chemical cleaning membrane as shown in figure 2.
3. Result and Discussion

3.1. Characteristic of Acid Mine Drainage

Acid Mine Drainage (AMD) as samples taken from Kintap, South Kalimantan-Indonesia was treated using pervaporation of membrane silica-pectin. Firstly, AMD was characterized with parameters which shown in table 1 and integrated with Indonesian regulatory standards regarding AMD for hygiene sanitation:

| No. | Parameters            | Unit   | Value | Standard Indonesia's Ministry of Health No. 32 2017th |
|-----|-----------------------|--------|-------|------------------------------------------------------|
| 1.  | Fe                    | mg/L   | 3.69  | 1                                                    |
| 2.  | Total Dissolved Solid | mg/L   | 652   |                                                      |
| 3.  | Conductivity          | µS/cm  | 1400  |                                                      |
| 4.  | pH                    | -      | 4     | 6.5-8.5                                              |

3.2. Morphology and Characterisation of Silica-pectin Membrane

Acid Mine Drainage (AMD) as samples taken from Kintap, South Kalimantan-Indonesia was treated using pervaporation of membrane silica-pectin. Firstly, AMD was characterized with parameters which shown in table 1 and integrated with Indonesian regulatory standards regarding AMD for hygiene sanitation:

3.2.1. Scanning Electro-Microscopy Membrane. The structure of the silica-pectin membrane was analysed using Scanning Electron Microscopy (SEM) which shows on figure 3. Analysed SEM aims to determine the surface morphological structure of the membrane. The surface area of the membrane affected the performance of membrane infiltration process [13, 34].
The thickness of membrane silica-pectin was shown in figure 3(a) the thickness of membrane in this study ~2 μm. The membrane with thin film was thinner result higher of water flux [7, 35–37]. In this study, the silica-pectin membrane using interlayer free membrane to reduce the thickness of membrane and to low cost in production. Therefore, in this work dip-coating of membrane silica-pectin was carried out 4 times to result 4 layers. Figure 3(b) shows the surface area of membrane silica-pectin. It shown there are no cracks on surface area membrane. The membrane silica-pectin looks asymmetrical due the calcination technique. In this work, calcination process using RTP method. There is an instantaneous temperature increasing during the calcined process [38, 39]. This technique has the advantage of speeding up the manufacture of membranes with still good results.

3.2.2. Fourier Transform Infra-Red. Silica-pectin membrane was analysed using FTIR-ATR technique. Figure 4(a). Shows region the FTIR-ATR spectra calculated peaks at 1300-700 cm\(^{-1}\). Peaks of siloxane (Si-O-Si) region of silica-pectin membrane measure on region 1080 cm\(^{-1}\), silanol (Si-OH) on region 976 cm\(^{-1}\) and pectin on region 790 cm\(^{-1}\). Region ± 970 cm\(^{-1}\) on the deconvolution of silanol peaks identified as the spin out mode of silica-carbon and shaking mode of carbon couple to Silica (Si) [15, 40–43]. Peaks at region 1080 cm\(^{-1}\) are delegated to a symmetric bond spin out vibration of Si-O-Si network [44]. In region 2001-2029 cm\(^{-1}\) there are the carbon bond vibration. The deconvolution of the band on region 976 cm\(^{-1}\) and region 1080 cm\(^{-1}\) to investigated of pectin with systematic incorporation measured using the peaks area ratio of Si-OH group vs Si-O-Si group was shown in figure 4(b). Fityk software was used to evaluate the amounts of areas on the silica-pectin membrane. The lowest peak area ratio between silanol and siloxane at xerogel sample on the membrane with concentration pectin 0.1 % calcined in air 400 °C. High concentration of siloxane region with an area of silanol region representing mesoporous or micropores [12, 13]. Actually, silica-carbon group contributed to determines the pore on the membrane silica-pectin. carbon chain clusters precedence to the formation of small pores but strengthen Si-OH bonds. This work results, the membrane concentration 0.5 % has high peak area ratio between silanol and carbon chains [45].
3.3. Performance of membrane after chemical cleaning process

The performance of silica-pectin membrane after chemical cleaning process of the membrane was shown in figure 5.

Generally, the membrane was cleaned due to membrane fouling which caused particles adhering to the membrane surface. Irreversible type of membrane fouling can be removed by physical washing [46]. However, particles fouling too attached the membrane surface, physical cleaning not optimal. Hence, chemical cleaning method of membranes can cleaning the membrane optimally and improve the flux and life time the membrane longer period [35, 47]. Therefore, this work using titanium dioxide such as chemical material in chemical cleaning membrane. Figure 5 was shown the performance of silica-pectin membrane with variations temperature of AMD (25 - 60 °C). Result of this work showed performance of silica-pectin
membrane after chemical cleaning using titanium dioxide is excellent. The flux recovery after chemical cleaning is higher than before cleaning process. Flux recovery from silica-pectin membrane concentration 0.1 % result 9.82 kg.m⁻².h⁻¹ with salt rejection 99.61 %. Then, flux recovery on silica-pectin membrane was 27.20 % higher before cleaning. The best result in this work is still flux recovery from membrane with concentration of pectin 0.5 %. The value of flux recovery during the pervaporation process 20 minutes is 17.54 kg.m⁻².h⁻¹ with salt rejection <99 % at room temperature. This flux showed that flux recovery of silica-pectin membrane recovery 65.32 % higher before cleaning. In other results, at temperature variations also showed that backwashing membrane with chemical cleaning was able to recovery water flux membrane. On feed temperature of 40 °C because of the driving force of the feed water temperature increase a high flux recovery. The flux value come to 13.59 kg.m⁻².h⁻¹ with salt rejection reaching 99.60 % at 0.1 % pectin concentration. Meanwhile, membranes with a pectin concentration of 0.5 % were able to produce a flux recovery of 21.26 kg.m⁻².h⁻¹ with a salt rejection of 99.60 %. The feed water temperature to evaluate the membrane performance was also carried out at on operating temperature of 60 °C. The resulting flux recovery increased at pectin concentrations of 0.1 % and 0.5 % consecutive 18.79 kg.m⁻².h⁻¹ and 29.51 kg.m⁻².h⁻¹ with the ability of salt rejection results still 99.52 % and 99.31 %. This proves that the silica pectin membrane that has been backwashed with chemical cleaning process exactly may improves the performance of the silica pectin membrane, also, the membrane is able to maintain salt rejection <99 %. The results of high salt rejection after chemical cleaning also prove that photocatalytic irradiation + TiO₂ catalyst able to change the compounds/ pollutants attached to the membrane surface can be converted into simple compounds which reduce fouling on the membrane as evidenced by the high salt rejection in the pervaporation process using a silica-pectin membrane.

4. Conclusion
Chemical cleaning of silica-pectin membranes using a photocatalytic membrane + TiO₂ catalyst shows that the evaluation results with pervaporation on various temperature are very excellent. Result shown in this work produce flux recovery reached 27.20 % and 65.32 % higher than the water flux before backwashing process. It is at an operating temperature of 25 °C. The increasing of flux recovery feed water temperature variations resulted in an increase with the ability of salt rejection <99 % in all variations in the temperature of the feed water.

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