1st International Conference Eco-Innovation in Science, Engineering, and Technology
Volume 2020
http://dx.doi.org/10.11594/nstp.2020.0543

NST PROCEEDINGS

OPEN ACCESS

Conference Paper

Wetland Saline Water and Acid Mine Drainage Desalination by InterlayeFree Silica Pectin Membrane from Banan Peels

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ABSTRACT

Wetland water and acid mine drainage are available in South Kalimantan, Indonesia. However, "Wetland saline water (WSW)" phenomena occur in the wetland areas due to the seawater intrusion, this water which contains a high salt concentration is unsafe to be consumed. While acid mine drainage (AMD) pollution becomes an issue in the mining industry that impact human life and the environment. Salt particles could be removed by using a silica pectin membrane. Banana peel has a high pectin substance. Banana pectin (0.5wt% and 0.1wt%) was employed in silica and calcined at 300 and 400 °C. We demonstrate the silica pectin template's performance without interlayer for wetland water and acid mine drainage desalination. Membranes were developed through a sol-gel method with silica source deposited from tetraethyl orthosilicate (TEOS) and performed by pervaporation at room temperature (~25 °C). As a result, 0.5wt% banana pectin concentration at 300 °C exhibited excellent performance with the highest water fluxes are 8.4 and 10.4 kg m^{-2} h^{-1} for WSW and AMD, respectively. Nevertheless, both membranes achieved high salt rejections up to 92%. Thereby, banana pectin as a carbon source impacts the stronger silica bond.

Keywords: Acid mine drainage, desalination, silica pectin membrane, wetland saline water

Introduction

Poor water quality caused by seawater intrusion and mine activities may affect the aquatic and human living. In South Kalimantan, Indonesia the massive mining industries play important roles in the economy. However, acid mine drainage (AMD) is formed as their product. During the rainy season, the water is mixed with coal at the mined coal and may run off to surface water (Nurofiq *et al.,* 2016). On the other hand, wetland saline water (WSW) in South Kalimantan has become a serious problem that should be solved. WSW salinity is even reached 40,000 ppm (3.5wt%) (Rampun, Elma, et al., 2019b). This value is found to be worst in the dry season because the water gets evaporated, and more salt remains in the water. Hence, desalination by pervaporation can be applied.

In principle, pervaporation is the separation of the mixture process by partial vaporization, which involves membrane (non-porous or porous). To apply low pressure, a vacuum pump is required (Rampun *et al.*, 2019b, Van der Bruggen & Luis, 2015). The pervaporation driving force is worked through chemical potential related to the concentration gradient between phases side by side of the interfacial barrier

How to cite:
Elma *et al.,* (2020). Wetland saline water and acid mine drainage desalination by interlayer-free silica pectin membrane from banana peels. *1st International Conference Eco. Innovation in Science, Engineering, and Technology*. NST Proceedings. pages 271-279. doi: 10.11594/ nstp.2020.0543

(Crespo & Brazinha, 2015). Various membrane-type have been used in pervaporation such as silica (Rahma *et al.*, 2019a), aluminum-rich zeolite beta incorporated sodium alginate (Adoor *et al.*, 2008), and polydimethylsiloxane (Kaddour *et al.*, 1998).

Among all types of membranes, silica has excellent mechanical strength and thermal stability. The unique characteristic of silica materials are physiochemical properties which consist of cross-linked network structure (Diniz Da Costa, 2000, Ellis, 2004), high surface area (Elma *et al.*, 2019), and vigorous (Elma, Riskawati, Marhamah, 2018). Owing to these properties, silica has been gaining widespread attention in material research.

Silica precursors are mostly manufactured from chemical precursor such as tetraethyl orthosilicate-(TEOS), methyltriethoxysilane (MTES), tetra ethyl vynilsilane (TEVS), or bis(triethoxysilyl)ethane (BTESE) (Ibrahim *et al.*, 2017), and ethyl silicate-40 (ES-40) (Maimunawaro *et al.*, 2020). These material sources obtain from an inorganic multipurpose chemical compound that is naturally found in quartz, sand, or flint (Moreth *et al.*, 2014). Each precursor can control and tune the silica network. Silica is an important material in thin-film fabrication. Sol-gel is one of the methods that often be utilized to fabricate silica membranes because of simplicity. In the sol-gel process, the hydrolysis and condensation are occurred by reacting alkoxide in an aqueous solution (Elma, Assyaifi, & Hairullah, 2017). Silanol (Si-OH) is created through a sol-gel process by employing acid catalysts at hydrolysis reaction, thus produce pore sizes less than 1 nm in silica thin film (Diniz *et al.*, 2002). Silanol groups will act as a binding site with the water presence, inducing silica matrices collapse and drop the salt rejection in membrane performance because of pore enlargement (Elma *et al.*, 2020; Elma *et al.*, 2020; Elma & Saputro, 2020, Elma *et al.*, 2015; Rahma *et al.*, 2020b).

To improve the silica membrane strength, several experiments have been done included the organic and inorganic combination materials (Mir *et al.*, 2018), incorporating metal oxide (nickel (Darmawan *et al.*, 2016, Darmawan *et al.*, 2017, Kanezashi & Asaeda, 2006), cobalt (Liu *et al.*, 2015; Smart *et al.*, 2012) and iron (Darmawan *et al.*, 2015), and carbon template silica (Lestari, *et al.*, 2020, Elma *et al.*, 2020a; Maimunawaro *et al.*, 2020, Rahma *et al.*, 2020a; Syauqiyah *et al.*, 2019). A recent study has shown carbon from pectin of apple peels replaces the use of synthetic carbon as a soft template. It resists the silica matrix collapsing and changed the structure to be more stable for pervaporation application (Pratiwi *et al.*, 2019a; Rahma *et al.*, 2019a; Rampun *et al.*, 2019a). The banana peel was investigated contain a high yield of pectin content until 24.8% (Khamsucharit *et al.*, 2018). It can be promising eco-friendly carbon. Therefore, in this work, we investigated the performance of interlayer-free carbon template silica membranes for wetland saline water and acid mine drainage desalination.

Methodology

Chemicals and materials

Wetland saline water (WSW) was collected from Muara Halayung village, Indonesia. Acid mine drainage (AMD) taken from Kintap South Kalimantan, Indonesia. Silica pectin sol was prepared by using tetraethyl orthosilicate (TEOS, 99,0%, Sigma-Aldrich) as silica precursor, ethanol (EtOH, 70%), dilute nitric acid (0.0008 M HNO_3, Merck), ammonia (0.0003 M NH_3, Merck), pectin from the banana peel, glycerol (85%, Merck), and aquadest. For membrane support, this study using a macroporous alumina tubular support membrane (Ceramic oxide fabricators, Australia) with pore size ~100 nm.

Membrane synthesis and characteristic

Firstly, TEOS mixed with EtOH under stirring at 0 °C for 5 minutes. Secondly, HNO_3 was added into solution and refluxed for 1 h at 50 °C, following by dropwise NH_3 under stirring for 2 h at the same condition. Then checked pH of pure silica sol until 6. Pectin dissolved into 5 ml of glycerol and then stirred 360 rpm for 95 minutes at 50 °C. Finally, pectin from the banana peel (0.5 and 0.1wt%) mixed onto the conducted silica sol for 45 minutes at 0°C.

Dipped macroporous alumina support membrane into silica-pectin sol to creates a thin film. Membrane dip-coating schematic process shows in Fig..1. All membranes variation were calcined in the air by a furnace at 300 °C for 0.5wt% of pectin and 400 °C for 0.1wt% of pectin. Repeat the dipping process and membrane calcined cycle for 4 times. The silica-pectin membranes morphology and thickness were examined using scanning electron microscopy (SEM) tested (Zeiss EVO LS15).

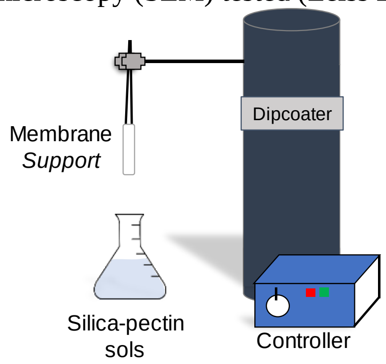

Figure 1. Dip-coating process schematic for silica-pectin membrane fabrication

Membrane pervaporation

The Pervaporation process was operated as shown in Fig. 2 which tested under room temperature (~25 °C). To keep away from concentration polarization, a peristaltic pump was used. Water flux F ($kg.m^{-2}h^{-1}$) was measured according to the Eq. (1):

$$F = \frac{m}{(A.\Delta t)} \qquad (1)$$

Where m is a permeate mass (kg) accumulated in the cold trap, A is the surface area (m2) of the membrane and Δt is time operation (h). Salt rejection R (%) was obtained based on Eq. (2):

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

Where Cf is the salt concentration (wt%) in the WSW and acid mine drainage water before treatment and Cp is a salt concentration in water permeate (wt%) after treatment. It was determined by a conductivity meter (OHAUS) correlated to the conductivity of retentate and permeate.

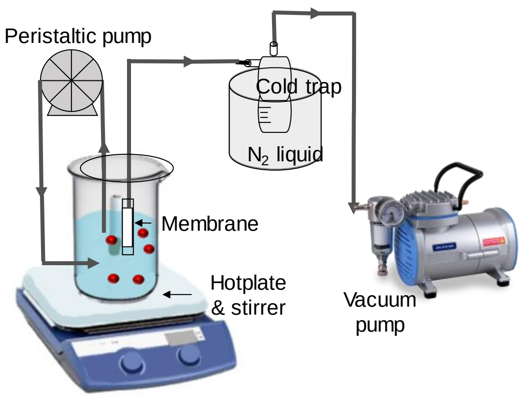

Figure 2. Pervaporation set-up by silica-pectin membranes for water desalination

Result and Discussion

characteristic of wetland saline water and acid mine drainage

Wetland saline water (WSW) and acid mine drainage (AMD) were treated using desalination by silica-pectin membranes. Generally, wetland water and AMD have bad qualities due to its water characteristics. The characteristic of WSW and AMD were displayed in Table 1. Initial WSW and AMD before treatment have pH below 7. AMD is known as pH value as low as −3.6 (Plant *et al.*, 2003) and containing high numbered of iron. Furthermore, both feed water containing a high concentration of salt above brackish water (0.3% NaCl). The concentration of NaCl in WSW and AMD is 10000 and 500 ppm, successively (Table 1).

Table 1. Characteristic of the wetland saline water and acid mine drainage in South Kalimantan, Indonesia

Parameter	Result		Unit
	Wetland saline water	Acid mine drainage	
pH	6.67	4	-
Fe	N/A	3.69	mg/L
Conductivity	9350	652	µS/cm
NaCl	10000	500	ppm
TDS	5400	324	mg/L

Membrane morphology

Silica-pectin membrane morphology from the banana peel is represented by scanning electron microscopy (SEM) image. The silica membranes by carbon induced by pectin 0.5% and 0.1% were obtained asymmetric structure illustrated on Fig. 3. Moreover, the surface area of silica-pectin membranes points out bearish or not smooth at all because no interlayer on membrane structure or called interlayer-free. Another reason, it also depends on the factor of the calcination technique (Pratiwi *et al.*, 2019b). Pore size differences between alumina support and a silica-pectin coating layer formed the rough surface on the membrane top layer (Elma, Hairullah, Assyaifi, 2018). RTP technique conducted without ramping rates (1 °C/min) bring out sudden temperature rise and creates thermal stresses on membranes. Although no crack and no defect are shown on the membrane surface. Other than that, silica-pectin membranes fabrication prepared by RTP technique is way faster than conventional techniques CTP (Pratiwi *et al.*, 2019c, Rahma *et al.*, 2019b).

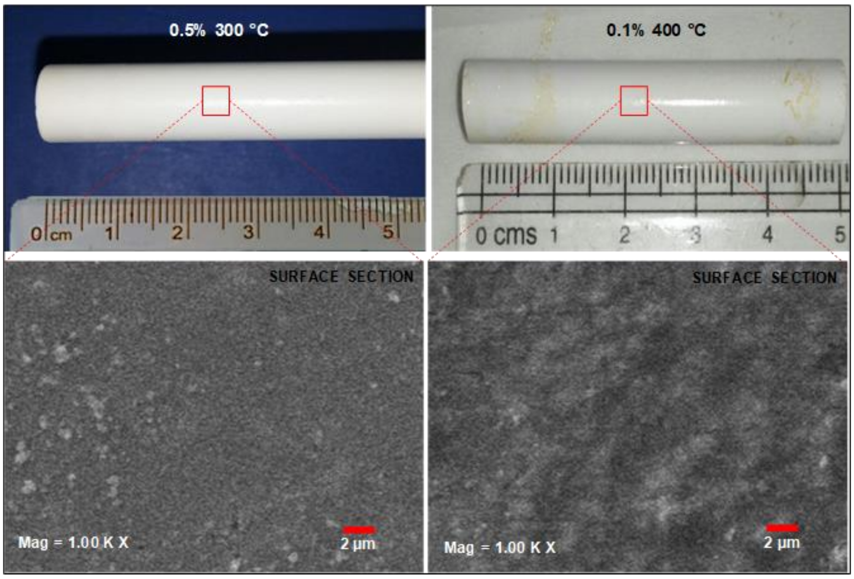

Figure 3. Surface sectional SEM images of silica-pectin membranes (left) 0.5 % pectin calcined at 300 °C (right) 0.1% pectin calcined at 400 °C.

The calcination technique contributes to membrane thickness. As shown in Fig. 4, cross-section SEM images of silica-pectin membranes exhibited the thickness of the silica-pectin top layer. Both of templating pectin of 0.5% and 0.1% into sol-gel was achieved thickness <2 μm. The calcined temperature also assisted on silica-pectin layer thickness. Silica-pectin membrane sintered over 300 °C becomes thinner as displayed in Fig. 4. Solvent and water trapped in silica matrices and then evaporated at 400 °C, after that thin layer was created (Elma *et al.,* 2019, Elma *et al.,* 2012, Lestari, *et al.,* 2020, Pratiwi *et al.,* 2019c, Rahma *et al.,* 2019b, Rampun *et al.,* 2019a). The silica-pectin membrane from bananas peel in this work thicker compare to pectin from apples which resulted <1 μm thickness (Rahma *et al.,* 2019b).

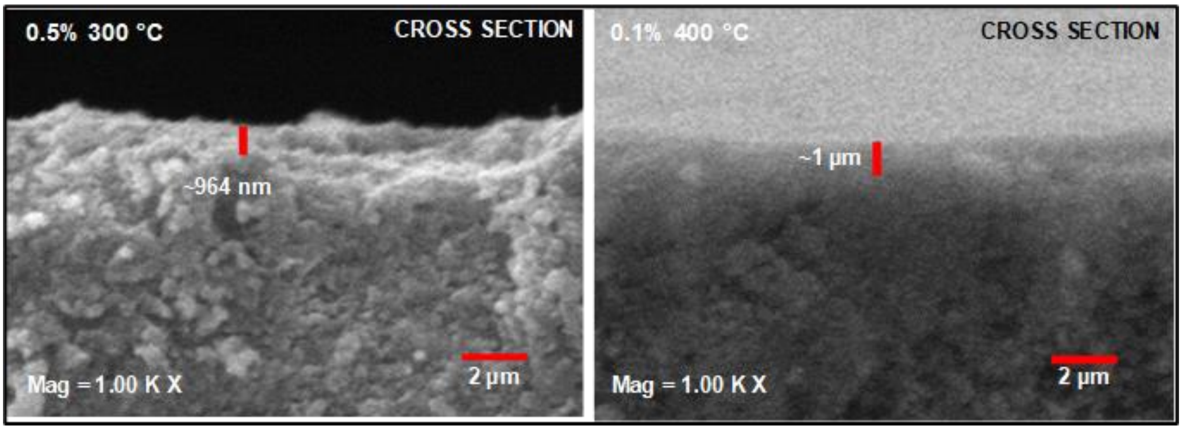

Figure 4. Cross sectional SEM images of silica-pectin membranes (left 0.5 % pectin calcined at 300 °C (right) 0.1% pectin calcined at 400 °C.

Membrane performance for desalination of wetland saline water and acid mine drainage

Silica-pectin membrane from banana peel via pervaporation performed using 2 varied feed i.e. WSW and AMD. Both of Fig. 5A and 5B represent the highest to smallest water flux sorted such as AMD > WSW of feed at room temperature (~25 °C). These results are influenced by the presence numbered of salt and natural organic matter (NOM) in feed water. WSW consists of high salt concentration and NOM, which lead to membrane need extra performances over AMD as feed. Moreover, NOM plays a role in blocking membrane pore and convey membrane performance become heavy, so the water flux going down (Goh *et al.,* 2018, Rahma *et al.,* 2019b). On the other hand, increasing the salt concentration will decrease the thermodynamic activity of water, so does the water flux (Wang *et al.,* 2016).

Attractively, carbon templated silica membrane by loaded of 0.5% pectin calcined at 300 °C shows the highest water flux of AMD of 10.8 $kg.m^{-2}.h^{-1}$. Whereas the lowest permeate flux was produced from WSW as feed at room temperature ~25 °C by adding pectin template 0.1% calcined at 400 °C. Increasing of carbon concentration for templates tend to form micelles and precipitate if in excess on silica sol-gel (Raman, Anderson, & Brinker, 1996). In this case, addiction of pectin of 0.1% and 0.5% into silica sol-gel was obtained water flux increase as well. This was also mentioned in Elma *et al.* (2015) which used variations of NaCl concentration (0.3-7.5% NaCl) as the feed. The high concentration of contaminants in membrane feed water results in concentration polarization which hurts reducing membrane performance (Elma *et al.,* 2012).

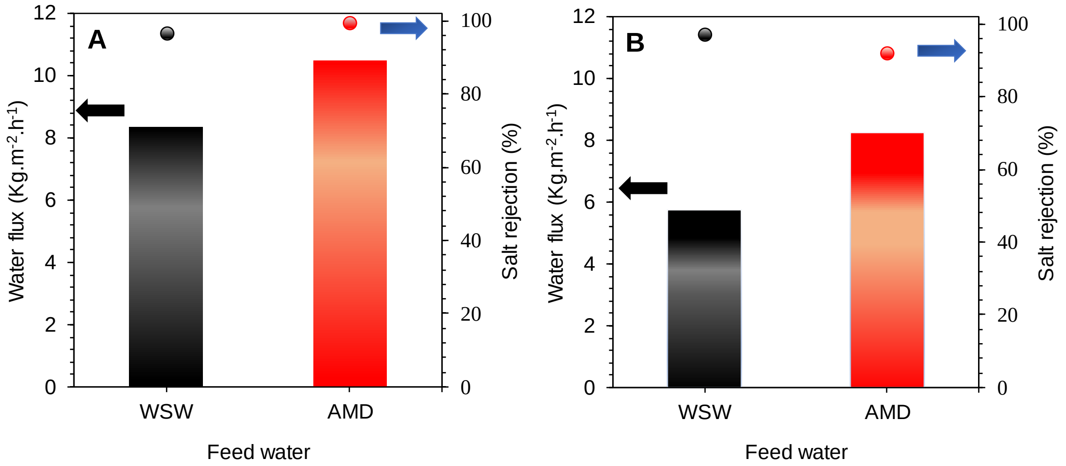

Figure 5. Performance of silica-pectin membranes for desalination of WSW and AMD (A) 0.5% pectin calcined at 300 °C (B) 0.1% pectin calcined at 400 °C.

Rejection of salt for all feedwater showed very well at all pectin loading (0.1-0.5 %). Based on Fig. 5A, salt rejections in all feeds are high >98 % for pectin 0.5% calcined at 300 °C. However, salt rejection of pectin templates of 0.1% in silica membrane resulted slightly lower of above 95% (Fig. 5B). The rejection of WSW is very good even though the silica-pectin membrane containing foulant which is the presence of a layer of humic compounds. According to Kim *et al.* (2009) organic fouling can increase or decrease salt rejection depending on the characteristics of the membrane used.

The membrane in this work presented approvingly as compared to previous carbon template silica membranes reported in the literature assorted in Table 1. For similar testing condition (WSW as feed; 25 °C), it is observed the banana pectin membrane in this work (8.3 $kg.m^{-2}.h^{-1}$; >98%) delivered a water flux 7 fold higher than pure silica membrane (1.19 $kg.m^{-2}.h^{-1}$; 85%) (Elma, Hairullah, & Marhamah, 2018), and at least 5 times high compare P123 template membrane (1.67 $kg.m^{-2}.h^{-1}$; 96%) (Elma *et al.*, 2018) and also 2 fold time than apple pectin membrane (4.45 $kg.m^{-2}.h^{-1}$; 99%) (Rahma *et al.*, 2019b), whilst salt rejection were high and comparable.

Table 2. Performance of silica base membrane for desalination

Membrane	Condition testing	Feed	Flux ($kg.m^{-2}.h^{-1}$)	Rejec-tion (%)	Ref
pectin banana peel	RTP, 25 °C(0.1&0.5% of pec-tin)	WSW	5.7-8.3	>98	This work
pectin banana peel	RTP, 25 °C(0.1&0.5% of pec-tin)	AMD	8.2-10.8	>95	This work
pectin apple peel	RTP, 25 °C (0.5% of pectin)	WSW	4.45	99	(Rahma, Elma et al., 2019b)
P123	RTP, 25 °C	WSW	1.25-1.67	66-96	(Elma, Fitriani et al., 2018)
silica	RTP, 25-60 °C	WSW	0.84-1.19	70-85	(Elma, Hairullah et al., 2018)

The purity of permeate water was assessed by evaluating the salt concentration calculated from a standard curve based on the measurements of conductivity. Figure 6 demonstrates that the permeate salt concentration was calculated to be 110-135 and 4-104 ppm for WSW and AMD feed solutions, respectively. These are well below recommended total dissolved salt concentrations of 600 ppm for potable water according to the World Health Organization. These results clearly show that the silica-pectin membranes prepared from the banana peel in this work can be used to treat wetland saline water and acid mine drainage at room temperature conditions with excellent performance.

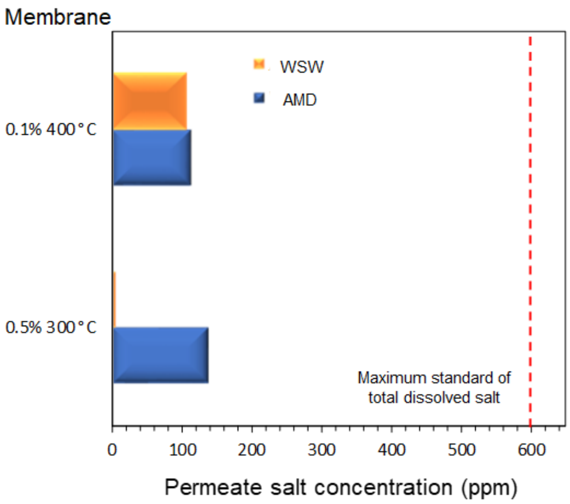

Figure 6. Permeate salt concentration as a function of the membrane silica-pectin concentration for desalination of WSW and AMD.

Conclusion

Silica-pectin membranes have been successfully developed by inducing pectin from banana peel into silica sol. Pectin concentration of 0.5% has outstanding performance for desalination. Silica-pectin membrane (0.5% pectin; 300 °C) delivered high water flux of 8.3 $kg.m^{-2}.h^{-1}$ (WSW) and 10.8 $kg.m^{-2}.h^{-1}$ (AMD). All salt rejection achieved over 98%. Moreover, the membranes can effectively produce potable water in these conditions because it reduces permeate salt concentration until below 600 ppm. Also, the silica-pectin membrane is favorable to be developed in wetland saline water and acid mine drainage desalination, considering that templating pectin as a carbon precursor to upgrade the silica membrane performance.

Acknowledgment

Authors thanks to M^2ReG (Material and Membranes Research Group) for the facilities provided. Muthia thanks to Applied Research Universities Grant 2019-2020, Thesis Magister Grant 2020, World Class Research Grant 2021-2022 the Directorate of Research and Community Service, the Ministry of Research and Technology/the National Agency for Research and the Innovation Republic of Indonesia, and Lambung Mangkurat University Research Grant 2020. Lambung Mangkurat University Grant 2020.

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