



Organo-Silica Membrane Prepared from TEOS-TEVS Modified with Organic-Acid Catalyst for Brackish Water Desalination

Aptar Eka Lestari^{1,2}, Muthia Elma^{1,2*}, Anna Sumardi^{1,2}, Zaini Lambri Assyaifi^{1,2}, Adi Darmawan³, Dwi Rasy Mujiyanti⁴, Isna Syauqiah¹, Aulia Rahma^{1,2}, Linda Suciwati^{1,2}, Yanti Mawaddah^{1,2}

¹Chemical Engineering Departement, Engineering Faculty, Lambung Mangkurat University (ULM), JL. A. Yani KM 36, Banjarbaru, South Kalimantan 70714, Indonesia

²Materials and membranes Research Group (M²ReG), Lambung Mangkurat University, JL. A. Yani KM 36, Banjarbaru, South Kalimantan 70714, Indonesia

³Departement of Chemistry, Diponegoro University, Semarang 50275, Indonesia

⁴Chemistry Departement, Mathematics and Science Faculty, Lambung Mangkurat University (ULM), JL. A. Yani KM 36, Banjarbaru, South Kalimantan 70714, Indonesia

*E-mail: melma@ulm.ac.id

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Abstract

A Silica membrane has a low hydro-stability. In this study, tetraethyl ortho silicate (TEOS), triethoxy vinyl silane (TEVS) precursors, and citric acid (as a catalyst) were blended to produce a mesoporous size of silica with a strong matrix for desalination process. The organo-silica membrane was calcined at 350 °C for 1 hour using a rapid thermal processing (RTP) calcination technique under vacuum to prevent the decomposition of carbon in the silica matrix. The membrane was dip-coated 4 times to obtain 4 layers. Fourier-transform Infrared Spectroscopy (FTIR) test was carried out to analyze the functional groups on xerogel, namely silanol, siloxane, and carbon. Membrane performance is operated by desalination through pervaporation using 0.3% NaCl feed water with variations in feed temperature. The permeate flux of water value was increased from 6.1 kg.m⁻².h⁻¹ to 12.1 kg.m⁻².h⁻¹ by increasing the feed water temperature from 25 to 60 °C. Meanwhile, salt rejection obtained on all membranes was above 99.0 %. The organo-silica membrane is suitable when applied to the desalination process through pervaporation.

Keywords: Mesoporous Organo-silica; TEOS; TEVS; Citric Acid; RTP; Vacuum Calcination

1. Introduction

A membrane technology membrane is a part of thermal technology that requires low energy to produce a water flux (Darmawan et al., 2017). A silica membrane has a good filter molecular properties. A fabrication of silica made through sol-gel has got attention because of the simplicity. The silica structure has pore sizes in the range 3-5 Å, it allows a water molecule passed the pore (dk = 2.6 Å), but inhibiting the passage of hydrate salt ions (e.g., Na⁺: dk = 7.2 Å dan Cl⁻: dk = 6.6 Å). In principle, the membrane separates the salt contains in water with the mechanism of filtering the molecular. Membranes are usually utilized in the desalination process via pervaporation.

Pervaporation is a desalination technology to produce a clean water with relatively low energy consumption compared to reverse osmosis membranes but still produces relatively a high flux (Kaminski et al., 2018; Rahma et al., 2019). Pervaporation has

offered several advantages such as high selectivity, low operating costs, can be operated at low pressure and high temperature, and offers a higher molecular sieving compared to zeolite and organic membranes (Mahmud et al., 2020; Syauqiyah et al., 2019). The pervaporation process generally uses an-organic membrane made from silica (Rahma et al., 2019).

However, silica membranes have a low hydro-stability. It would not be good to be applied in long-term performance (Elma M. et al., 2019). The contact between the silica membrane and water causes the pores of the membrane to collapse over time (pore collapse) This is due to the hydrophilic nature of silanol contained in silica (Rampun Erdina L. A. et al., 2019). A membrane modification is needed to prevent it and increases the silica strength as well (Elma Muthia et al., 2020a; Elma Muthia et al., 2020c). Several studies have been developed to improve the performance of silica membranes such as hybrid organic-inorganic materials,

incorporation of metal oxides into the silica network (BP et al., 2011; Duke et al., 2007; Maimunawaro et al., 2020).

Previous research has reported a rapid thermal processing (RTP) under vacuum condition of calcination resulting in a better structure of the organo and carbon templated silica membrane. The vacuum condition maintains the mass of organic carbon to be not easily decomposed. Yang et al. (2017) has shown a prepared membrane with vacuum calcination only slightly lost mass, namely 3.5% by weight, while the weight loss of 9.5% mass happened for the membrane calcined in N₂. Elma Muthia et al. (2015b) reported the vacuum calcination producing in more mesoporous silica matrix and the resulting carbon is higher than N₂ calcination. It gives which results in relatively superior performance during the desalination process.

Carbon sources can be obtained from the addition of catalysts or precursors (Yang et al., 2017). In this study, a dual precursors, namely tetraethyl ortho silicate (TEOS) and triethoxy vinyl silane (TEVS) were used to produce a silica matrix with the mesoporous size. Wahab et al. (2004) has obtained a hydrophobic micropore silica from a mixture of that TEOS/TEVS as precursors and HNO₃ as catalyst that resistant to humid atmospheres. Whereas Elma Muthia et al. (2015b) used the TEOS/TEVS/KPS precursors with acid (nitric acid) and alkaline (ammonia) catalysts to produce the mesoporous silica. The TEVS has a double bonds which can be hydrolyzed and polymerized to form a single C-C bonds sequentially to fill more space in the porous structure. TEVS is a silica precursor that has carbon chain and can be used to form Si-C bonds. Yang et al. (2017) studied the combination of TEOS/TEVS/P123 with acid (nitric acid) and base (ammonia) catalysts produced in a good performance for desalination process. The TEOS precursor does not have ligands, whereas TEVS has a ligand template based on the vinyl group. TEVS has previously been used to form the micropore structures on a porous substrate interlayer. Therefore, a pure TEVS is not suitable for producing amorphous silica because the functional groups form a block in the pore (Elma Muthia et al., 2015a).

In desalination process, the mesoporous silica is needed to design through the addition of the catalyst. Hence in this study, TEOS and TEVS precursors were employed with citric acid addition. The citric acid has a C (carbon) group to believe in strengthen the silica matrix. In addition, the calcination process is

carried out by vacuum calcination to maintain its carbon mass. According to previous research (Elma Muthia et al., 2015b), the water flux value will be increase as feed water temperature increases, led to achieve a good performance. Therefore, this study using a variation of the feed water temperature (25°C, 40 °C dan 60 °C) to investigate the water flux and salt rejection in TEOS/TEVS membrane with the citric acid addition.

2. Method

2.1. Materials

The materials used in this study consisted of tetraethyl ortho silicate (TEOS, 99.0%, GC, Sigma-Aldrich) and triethoxy vinyl silane (TEVS) (97.0%, Sigma-Aldrich) as a precursor to silica, ethanol (EtOH: 70%) and water as a solvent, dilute citric acid (23.4%, Merck) as a catalyst and macroporous alumina substrates α Al₂O₃ tubular support (Ceramic Oxide Fabricators, Australia) with an average pore size of 100 nm. The molar ratio used is TEOS: TEVS: EtOH: H₂O: The citric acid is 0.9: 0.1: 38: 5: 0.1.

2.2. Preparation of Organo-silica Sol and Membrane

The sol and membrane of organo-silica preparation procedure were referred to the previous studies by Elma Muthia et al. (2020b). The fabrication of organo-silica membrane was divided into two main steps, i.e.; (1) synthesis of organo-silica sol; and (2) dip-coating of membrane organo-silica.

2.2.1 Synthesis of organo-silica sol

Sol gel organo-silica was made by mixing TEOS, TEVS and EtOH precursors into a Schott bottle with 0 °C operating conditions. The solution was then stirred using a magnetic stirrer at 360 rpm. Furthermore, the citric acid catalyst was added and reflux at a temperature of 50 °C for 3 hours. Then, the pH value of silica sol is measured. The organo-silica sol was dried in an oven at 60 °C for 24 hours. The dried soles, which called xerogels, were crushed using a mortar and pestle until become powder. The powder is then calcined using a vacuum furnace. Briefly, procedure of experiment can be showed in Figure 1. The xerogel was characterized by FTIR (Fourier-transform infrared spectroscopy) to determine its functional groups. The calcined organo-silica xerogel was placed on an optic window of FTIR equipment. Then, the infra-red (IR) were contacted through the sample which measuring absorbance to exhibits IR spectra

at wavelength number between 600-1400 cm^{-1} .

2.2.2 Dip-coating of Organo-silica Membrane

The Membrane was prepared by dipping the $\alpha\text{-Al}_2\text{O}_3$ tubular support membrane into the silica sol with withdrawal rates of 10 and 5 cm per minute and total immersion time of 2 minutes. The coated membrane was calcined via the RTP (Rapid Thermal Processing) method under vacuum condition for 1 hour at a temperature of 350 °C. After calcining, it is cooled for 30 minutes. This process is repeated 4 times to obtain 4 layers.

2.3. Desalination Experiment

The organo-silica membrane was applied for pervaporation. The feed water contained 0.3% NaCl. In desalination process, the temperature of feed water was varied (20, 40, and 60 °C) to investigate the influence of the temperature to the water flux and rejection (Elma Muthia et al., 2015b). Desalination process via pervaporation can be run in equipment installation as shown in Figure 2.

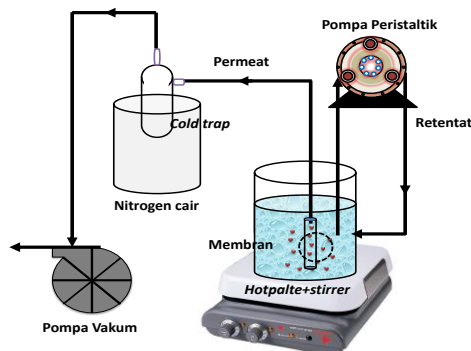


Figure 2. Desalination Process of 0.3% NaCl via Pervaporation Set-up

3. Results and Discussion

3.1. Characteristic of Organo-Silica Membrane

Organo-silica xerogel analyzed via FTIR to determine the functional groups of siloxane groups (Si-O-Si), silanol (Si-OH) and carbon silica (Si-C). The results of FTIR spectra test with a wavelength range between 1,400-600 cm^{-1} is shown in Figure 3. The peaks of the siloxane (Si-O-Si) groups were identified at wavelength 800, 1048 dan 1200 cm^{-1} . This results in line with previous studies that found the peaks of siloxane at 800, 1060 dan 1200 cm^{-1} (Elma Muthia et al., 2015b) and at wavelength 800, 1060 dan 1220 cm^{-1} (Elma Muthia et al., 2015a). The silanol group (Si-OH) was identified at a wavelength of 966 cm^{-1} . The appearance of silanol group in our work agrees with the study elsewhere (Elma Muthia et al., 2018) which is identified at a wavelength of $\sim 960 \text{ cm}^{-1}$.

The formation of silanol groups in the silica matrix promotes a small porous membrane (microporous), while the large number of siloxane groups causes a larger porous membrane (mesoporous). The siloxane group is mostly formed from condensation reactions, and hydrolysis reactions forming in the silanol groups (Neville & Seyfaee, 2013). The optimum conditions are indicated by the lowest Si-OH / Si-O-Si value. It shows the siloxane concentration is greater than silanol. Mesoporous sized membranes have the ability to separate the water and salt and good for application in the desalination process (Yang et al., 2017). Then, the silica carbon (Si-C) groups presence was identified at a wavelength of 796 cm^{-1} . Carbon compounds (CH_3) were identified to bind to silica compounds at a wavelength of 760-960 cm^{-1} .

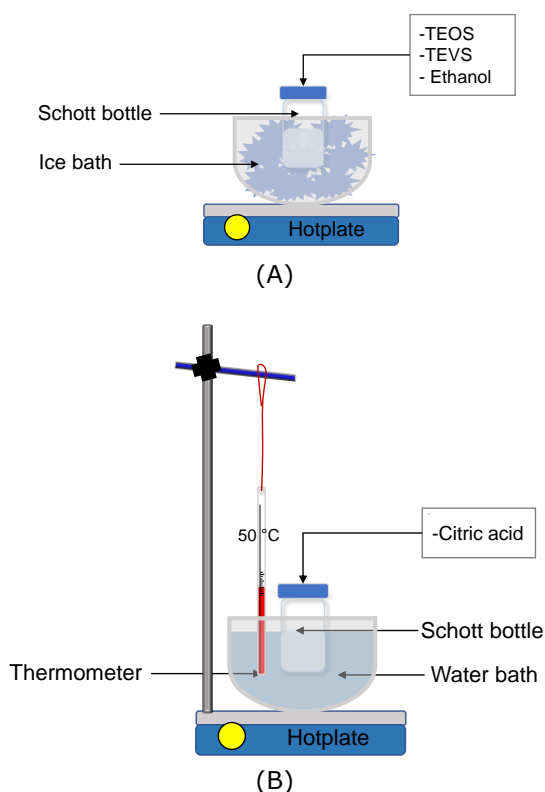


Figure 1. Illustrated of Organo-silica Sol Process by Sol-gel Method in ice bath (A), and in water bath (B)

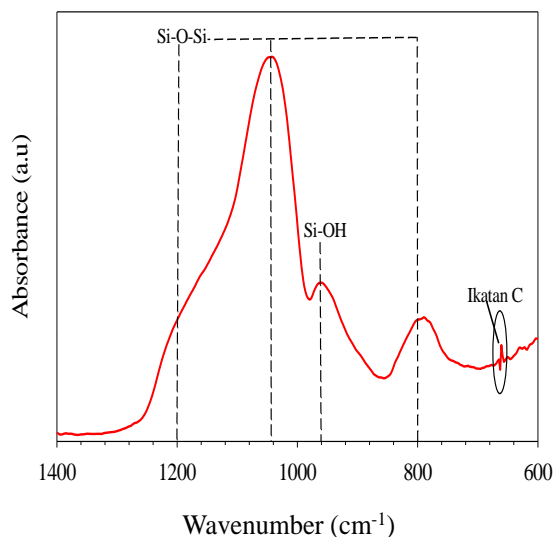


Figure 3. IR Spectra of Organo-silica Xerogel

This result is also similar to previous research reported by Pratiwi et al. (2019b), which is a carbon stretching vibration (Si-C) appeared from silica-organic at a wavenumber of $\sim 789 \text{ cm}^{-1}$. The Si-C bond was formed due to the addition of TEVS precursors (Elma Muthia et al., 2015a) and citric acid as a carbon source in the sol gel process. In comparison with previous studies the incorporating TEVS in TEOS precursors, could increase the Si-C bond (Elma Muthia et al., 2015a).

Our study has added a citric acid to maximize the silica matrix strength. The Si-C bond in the silica matrix has a low water adsorption rate on the surface of the carbon bond. It can increase the hydrophobicity of the membrane (Yang et al., 2017) and suitable for membrane coating (Darmawan et al., 2017; Saputra et al., 2018).

This is because the carbon bonds are stronger compared to silanol (Pratiwi et al., 2019b). To investigate further about the effect of TEVS and citric acid to silica structure, the deconvolution was performed using Fityk software as shown in Table 1. FTIR spectra data were performed with peak fitting using the Gaussian function with an error limit of $\pm 0.5\%$ and the on unit refers to Park (2013) and Saputra et al. (2018).

The combination of TEOS and TEVS precursors with a citric acid catalyst produces a low silanol value of 0.9289 cm^2 and high amount of siloxane until 12.0890 cm^2 and a carbon value of 2.2329 cm^2 . In comparison with other study that employed ES40 as silica precursor and P123 template as presented in Table 1, our study offered higher over 2-fold times siloxane and silica carbon of 2.2329 cm^2 . It proves the addition of TEVS precursor as silica source containing the carbon bonds can increase the Si-C bond. C-C single or bonds obtained from the hydrolyzed and polymerized double bonds in TEVS. This is in accordance with previous research (Elma Muthia et al., 2015a) that the combination of TEOS and TEVS can produce a mesoporous silica matrix. This is because citric acid can be diminishing the silanol groups and formed into stronger siloxane and silica carbon instead. The RTP calcination technique in this study requires 1 hour without leaning rates (cooling rate) unlike the CTP calcination. Research states the RTP calcination technique increasing the rate of condensation.

Several studies have reported that the organo-silica calcined by RTP using air condition of calcination produces a mem-

Table 1. The Resulting Peak Area Using Fityk Software

Xerogel	Area Ratio (cm^2)			References
	Si-OH	Si-O-Si	Si-C	
TEOS/TEVS/citric acid	0.9289	12.0890	2,2329	This work
TEOS/citric acid reflux 0°C	3.1059	9.4823	1.4701	(Lestari A. E. et al., 2020)
TEOS/citric acid reflux 50°C	2.2614	13.0394	1.7220	(Lestari A. E. et al., 2020)
TEOS/citric acid/ammonia	2.5400	11.6400	1.0600	(Lestari R. A. et al., 2020)
Pure ES40	0.6125	9.5806	0.3718	(Rahman et al., 2020)
Hybrid P123	0.0146	14.3494	0.7750	(Rahman et al., 2020)
ES40 P123 template	9.51×10^{-5}	1.0264×10^{-3}	8.10×10^{-5}	(Rahman et al., 2020)

brane with great performance (Elma Muthia & Setyawan, 2018; Mujiyanti et al., 2019; Pratiwi et al., 2019a; Rampun Erdina Lulu Atika et al., 2019; Syaunyah et al., 2019). Instead of the air, the vacuum condition in this research successfully preventing a high lost mass of organic carbon. Elma Muthia et al. (2015b) reported that vacuum calcination resulted in a more mesoporous silica matrix and higher carbon yield.

3.2. The Performance of Organo-Silica Membrane Performance

The performance of organo-silica membranes was tested in the desalination process of NaCl 0.3wt% with a variation of the feed water temperature is 25 °C, 40 °C dan 60 °C. Figure 4 represents the organo-silica membrane performance. It shows the water flux increases along with the increase of feed water temperature from 6,1 to 12,1 kg.m⁻²h⁻¹. At high feed water temperatures, the effect of increasing steam is also enhanced because of the driving force going up (Elma Muthia et al., 2015b). By thus, increasing evaporation system which is directly proportional to the steam generated (Duke et al., 2007; Xu et al., 2011). Pervaporation worked by evaporating the feed. Generally, the temperature gives an impact on permeate transport behavior and increase the vapor pressure difference. Meanwhile, the resulting salt rejection was dropped as feed water temperature increased from 99.72% to 99.23%. This indicates the

occurrence of a pore damage in the membrane because the temperature of the feed water is too high. A high feed temperature obtained a larger free volume in organo-silica membrane's pore. The higher water flux and salt rejection, it indicates a good membrane performance.

Not only temperature, the membrane structure (mesoporous). The higher feed water concentration can cause a fouling on the membrane and a concentration polarization which resulting in low membrane performance. Fouling could be clogging the membrane pores.

The performance of the organo-silica membrane in this study is not inferior to other membranes (Table 2). Yang et al. (2017) work using the P123 as an addition to the matrix bond in silica and has longer chain carbon than, TEVS precursors and citric acid in our study. Previous work utilizes the TEOS precursor, the water flux obtained in study showed the lower water flux compares to our study, but the salt rejection value remains good. This is because the act of TEOS and TEVS precursors. In addition, it can be seen that pure silica membranes tend to have larger pores. In addition, different calcination techniques also affect the performance of the resulting membrane.

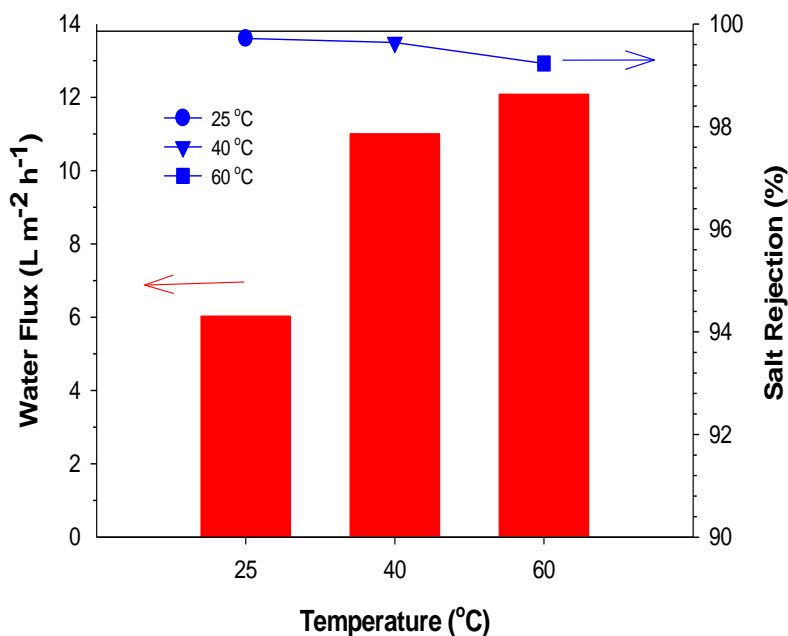


Figure 4. Water Flux dan Salt Rejection Organo-silica Membranes with Variations in Feed Temperature

Table 2. Comparison Performance of Membrane Variation Type for Water Desalination

Membrane	Calcination Method	Feed Temperature (°C)	Water Flux (kg.m ² .jam ⁻¹)	Salt Rejection (%)	References
Pure silica membrane	CTP (air)	25	6.8	98.2	(Elma Muthia et al., 2013)
Silica cobalt	CTP (air)	25	~5	>99	(Elma Muthia et al., 2015d)
P123 carbonized template silica	CTP (vacuum)	25	~2.5	>99	(Elma Muthia et al., 2015b)
Interlayer free hybrid silica-carbon TEOS/TEVS/P123	RTP (N ₂)	25	>25	98.5	(Yang et al., 2017)
Interlayer free hybrid silica-carbon TEOS/TEVS/P123	RTP (vacuum)	25	>25	98.6	(Yang et al., 2017)
Interlayer free membrane ES40	RTP (air)	60	17.8	>99	(Wang et al., 2016)
Organo-silica TEOS/TEVS/citric acid	RTP (vacuum)	25	6.1	99.72	This work
Organo-silica TEOS/TEVS/citric acid	RTP (vacuum)	40	11.2	99.64	This work
Organo-silica TEOS/TEVS/citric acid	RTP (vacuum)	60	12.1	99.23	This work

4. Conclusion

The conclusion of this study is that the combination of TEOS, TEVS as precursors and citric acid as a catalyst can produce mesoporous silica matrices. TEVS precursors increases the Si-C bonds that led to more robust membrane structure. The RTP calcination technique under vacuum will maintain the organic carbon mass to be not easily decomposed. The water flux increased as feed water temperature increased from 6,1; to 12,1 kg.m⁻².h⁻¹. Salt rejection decreased from 99.72% to 99.23%. Despite of the salt rejection flux dropped, but the membrane is suitable to be applied in desalination

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