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Abstract:

Clean water is essential source for household purpose. However, many surface water contain high salt concentration was found. In this work, membrane was made using tetraethyl orthosilicate (TEOS) as silica precursors and citric acid as single organo catalyst. Membranes were calcined at 200 and 250 °C using Rapid thermal processing (RTP). All membranes were tested via pervaporation. Pervaporation processes allow membrane to separate salt from water as vapour phase with vacuum condition required. Permeate was collected in the cold trap after condenses. This study focus to the performance of organo silica membrane in variance of refluxed 0 and 50 °C and feed concentrations (0.3, 3.5 and 5wt% NaCl) at room temperature (~25 °C). Optimum condition was obtained at reflux 50 °C with high siloxane and Si-C bonds. Carbon content from citric acid promote silica network more strength. The good performances in variance feed concentration were also showed at reflux 50°C with 0.3324 kg.m⁻².h⁻¹ (0.3 wt%), 0,2290 kg.m⁻².h⁻¹ (3.5 wt%) and 0.2168 kg.m⁻².h⁻¹ (5 wt%). These membranes are categorized as mesoporous and achieve excellent salt rejection >95%.

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Performance of Mesoporous Organo Silica Membrane for Desalination

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Keywords: Mesoporous silica membranes, organo catalyst, pervaporation.

Abstract. Clean water is essential source for household purpose. However, many surface water contain high salt concentration was found. In this work, membrane was made using tetraethyl orthosilicate (TEOS) as silica precursors and citric acid as single organo catalyst. Membranes were calcined at 200 and 250 °C using Rapid thermal processing (RTP). All membranes were tested via pervaporation. Pervaporation processes allow membrane to separate salt from water as vapour phase with vacuum condition required. Permeate was collected in the cold trap after condenses. This study focus to the performance of organo silica membrane in variance of refluxed 0 and 50 °C and feed concentrations (0.3, 3.5 and 5wt% NaCl) at room temperature (~25 °C). Optimum condition was obtained at reflux 50 °C with high siloxane and Si-C bonds. Carbon content from citric acid promote silica network more strength. The good performances in variance feed concentration were also showed at reflux 50°C with 0.3324 kg.m⁻².h⁻¹ (0.3 wt%), 0,2290 kg.m⁻².h⁻¹ (3.5 wt%) and 0.2168 kg.m⁻².h⁻¹ (5 wt%). These membranes are categorized as mesoporous and achieve excellent salt rejection >95%.

Introduction

The rapid grow human in world population over time make clean water is very necessary to be used. According to World Health Organization, the world's population have no access to potable water is more than 15% and does not have access to sanitation about more than 37%[1]. It can be seen that the water needed for consumption with the availability of clean water in the world is very unbalanced. Therefore, this clean water crisis needed to be overcome. Desalination can remove salt contain in water.

Desalination is one of the alternatives treatment that has been widely used in the world. Methods in desalination process include reverse osmosis (RO), membrane distillation (MD) and pervaporation (PV). In pervaporation method, desalination is carried out using the principle of phase change to separate water with salt particles in a vacuum medium. Pervaporation more economical compared to reverse osmosis method. Silica-based membrane is an attractive option for water desalination due to high selectivity [1].

Sol-gel method is widely used for membrane coating processes because it is very simple and cost effective routes, but still offers the flexibility to adjust the porosity required [1]. Citric acid is an organic acid catalyst and content carbon that is templated into silica network. This fabricated materials is applied for coated membrane layers (thin film) attached to membrane substrates. It is called organo silica membranes.

Controlling pH is an important way to adjust the pore size of membranes. The sols prepared in pH 6 have reported give the lowest concentration of silanol (Si-OH). It can improve hydrostability and optimizing conditions for film coating. Hence, pH 6 silica membranes delivered high water flux of 9.5 kg.m⁻².h⁻¹ (salt rejection 99.6%) and 6.8 kg.m⁻².h⁻¹ (salt rejection 98.2%) for the 0.3 and 3.5 wt % saline solutions, respectively [2].

This study was aimed to investigate the performance of organo silica membranes in various of refluxed 0 and 50 °C and feed concentrations (0.3, 3.5 and 5wt% NaCl) using sol gel process to synthesize the functionalization of organic catalyst in silica matrices.

Materials and Method

Materials. The materials used are *Tetraethyl orthosilicate* (TEOS, 99.0%, (GC) Sigma-Aldrich) as silica precursor, diluted citric acid 0.1 %, ethanol (99%), aquadest (H₂O), citric acid as catalyst solvent, membrane support (α -Al₂O₃), NaCl, and nitrogen liquid.

Material synthesis. Silica sols were synthesized by a single organo catalyst sol-gel process using citric acid. Firstly, 18.66 grams tetraethyl orthosilicate (TEOS, 99.0%, (GC) Sigma-Aldrich) was added drop-wise into 20 mL ethanol and stirred for 5 min under vigorous stirring to achieve a complete hydrolysis of the alkoxy groups in cold condition at 0 °C. Then followed by adding diluted citric acid of 0.1 % in variance of reflux temperature of 0 and 50 °C for 3 hours after added Ethanol of 140,96 mL to avoid partial hydrolysis. The organo silica sol which was then dried in oven at 60 °C for 24 h and the dried gel was obtained. Sol gel process set-up was show in Fig 1.

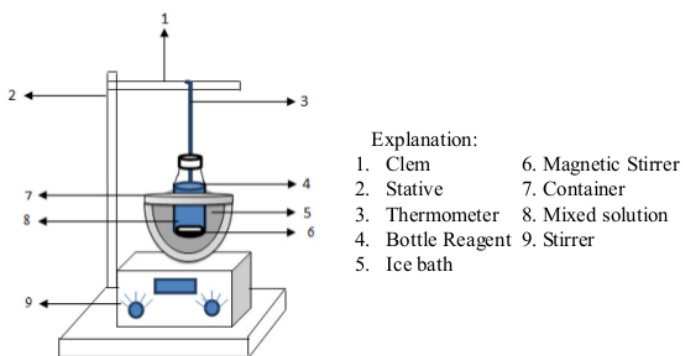


Figure 1. Sol gel process set-up

Preparation of xerogels. Organo silica sols was then placed in the petri dish and dried in the oven at temperature 60 °C for 24 hours. Then, organo-silica xerogels were grounded into powder. Lastly, xerogels were calcined at 250 °C using RTP technique for 1 hour without applying ramping/cooling rates.

Characterization of silica-carbon xerogels. FTIR (Fourier Transform Infra-Red) is used to investigate the functional groups of organo silica xerogel. The evolution of chemical structure, particularly the effect loading on the silanol to siloxane transition of the calcined silica matrices was analysed by the FTIR-ATR technique. FTIR spectra data were collected from FTIR type Bruker Alpha. Instrument type: alpha sample compartment RT-DLaTGS accessory: ATR platinum Diamond 1 Relf. The spectra were collected from a total of 30 scans ranging between wavelengths 500-4000 cm⁻¹.

Fabrication of organo Silica Membrane. Silica sol-gel were coated into α -Al₂O₃ support via dip-coating as shown at Fig 2. Dipcoater was settled up with down speed 5 cm/s and well time for 2 minutes. Calcined at 200 and 250 °C for 1 hour and cooled at room temperature. These steps (dipcoating-calcination) were repeated until 4 times to produce 4 thin layers.



Figure 2. Schematic of dipcoating process

Membrane performance. The membrane was assembled using pervaporation set up for desalination as shown in Fig 3. In this setup, membranes were tested in dead end mode. Membrane was connected to the cold trap (in liquid nitrogen) and vacuum pump. Organo silica membrane was immersed in the feed tank containing with varied sodium chloride (0.3, 3.5 and 5wt% NaCl). The feed solution was recirculated via a peristaltic pump where the retentate stream was fed back to the feed tank. Additionally, the feed solution was under constant stirring to minimise excessive salt concentration polarisation on the membrane surface.

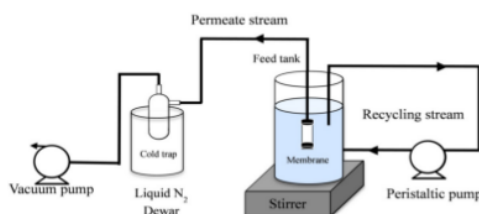


Figure 32. Pervaporation set-up for water desalination process

Results and Discussion

Silica sol was made by sol gel process using citric acid as single organo catalyst which produced silanol and siloxane groups. Citric acid have three carboxyl (-COOH) and one hydroxyl(-OH) groups.

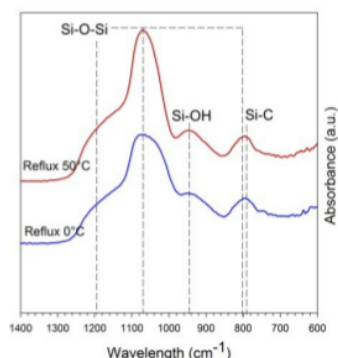


Figure 4. FTIR spectra xerogel calcined at 250°C

The results of FTIR test of xerogel samples in Fig 4 that shown the vibration bands in the region of 1400-600 cm^{-1} . Siloxane group (Si-O-Si) of both xerogels were detected in peak wavelengths at 1080 and 1200 cm^{-1} and 800 cm^{-1} , for xerogel in reflux temperature 0 and 50°C respectively. At wavelength 970 cm^{-1} related to Si-OH (silanol group) vibration [3]. As can be seen at wavelength 790 cm^{-1} is bending vibration of Si-C [4]. Through Fig 4, the siloxane peak at reflux 50°C is higher than reflux 0°C as well as the silanol and Si-C bondings happened due to a higher temperature (50°C) both of the condensation reaction will occur better than at lower temperature that tend to

stop the reactions as well [5]. The higher siloxane bridges and Si-C bonds were contribute to increasing the hydro-stability of the materials, in this work is membrane. As from Table 1 at 50°C of reflux temperature gives higher Si-C bonds and highest silanol groups was at 0°C of reflux temperature. Those results were convince that 50°C is the best condition of reflux temperature to maximize condensation reaction.

Fig 5 also reinforced previous data that shown the peak area ratio of Si-OH/Si-O-Si groups that carried out with Fytik software using Gaussian lineshapes with the least square routines [6]. Good result was obtained if the value of ratio is the lowest one that shown at 50°C of reflux temperature alcohol and water condensation will optimum occure and forming more siloxane bridges and new bonding of Si-C. High content of Si-C bondings will makes the silica matrices strengthen and robust that able to applicate in water desalination. If we see and focus to pH value in Table 1 the lower pH value was represented of 50°C as reflux temperature that means the best condition that obtained in this work [7]. However, that could be ignored due to the differences of pH value also 1 point (3 vs 4). In this work we used weak acid, but consist high carbon content that could increasing the hydro-stability and strengthen of silica matrices.

Table 1. Deconvolution Silanol/Siloxane

Xerogel	pH	Si-O-Si	Si-OH	Si-C	Si-OH/Si-O-S
Reflux at 0°C	4	9.48238	3.10597	1,4701	0.32755
Reflux at 50°C	3	13,0394	2,26148	1,7220	0.17343

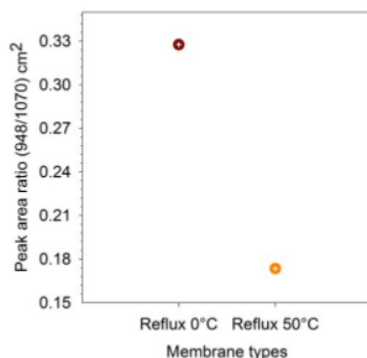


Figure 5. The peak area ratio at 948 cm^{-1} /1070 cm^{-1} for the spectra as a function of the reflux

To understanding surface area and pore size of organo silica, N_2 adsorption-desorption is investigated as shown on Fig 6. It shows the curve both of xerogel reflux 0 and 50°C are categorized as type IV H4. For xerogel refluxed at 0 °C, the hysteresis of N_2 adsorption-desorption starting occurred at relative pressure ~ 0.4 p/p°, and the hysteresis of xerogel refluxed at 50°C observed at ~ 0.1 p/p°. Table 2 is summarized more detail about surface properties. Reflux of 50 °C provides the lowest surface area, total pore volume and pore size compared to reflux of 0 °C. In addition lower reflux temperatures tend to form mesoporous material with loop hysteresis in the range 0.4-0.7 p/p° categorized as isotherm type IV.

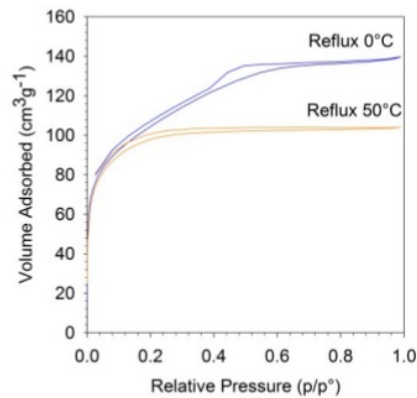


Figure 6. Mass loss curves of xerogel at reflux 0°C and 50°C

If compare the surface area and total pore value of both samples, reflux at 0°C gives a quite high value (just only 11% for surface area and 25% for total pore volume) than reflux at 50°C. Then the pore size of both samples were categorized into mesoporous [8]. This result prove that the single organo catalyst with high carbon content is a promising modification that could remove the catalyst uses (dual catalyst) and improving the strengthen of silica matrices.

Table 2 Surface properties of organo-silica xerogels

Membrane type	Surface area (m ² /g)	Total pore volume (cm ³ /g)	pore size (nm)
250°C calcination (reflux 0°C)	354	0.215	2.83
250°C calcination (reflux 50°C)	315	0.161	2.47

As an organic additive, citric acid has shown special abilities to creating amorphous mesoporous silica materials in the sol-gel process [9]. At reflux 0°C the result shows pore size bigger than at reflux 50°C which means that the ability in water desalination is better for silica thin films. Based on the Fig 7 can be seen the surface and thickness of the silica membrane for a calcination temperature of 250°C.

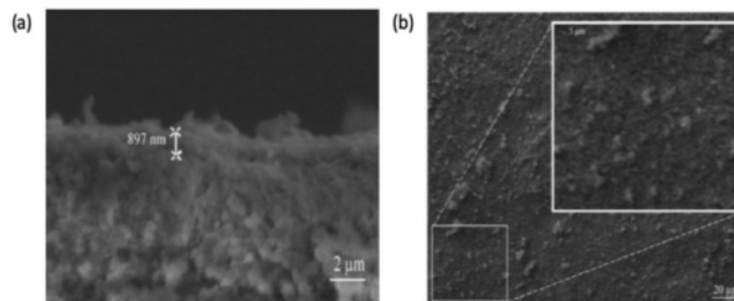


Figure 7. Image of SEM Analysis for Silica Membrane Calcined at 250°C, a) Cross Section, b) Surface Area

Fig 7.a shows the thickness of the thin layer and the macroporous α - Al_2O_3 substrate which is about ~ 897 nm, that indicates during the coating and calcination process the layer forms on the membrane surface. Whereas in Fig 7.b it can be seen that the membrane surface shows the presence of particles attached, this is because at a temperature of 250°C the solvent particles are still trapped in the membrane pores so that the surface looks rough. According to research [8] the previous membrane with a calcination temperature of 450°C shows a smoother surface area without roughness and boulders of the alumina particles protruding on the membrane surface. This is because the solvent particles have been evaporated, thus forming a smooth surface. The thickness of the thin layer on this membrane will affect the performance obtained.

At Fig 8 shows the performance membrane at reflux 0°C and 50°C on citric acid concentration is 0.1 % at 250°C calcination.

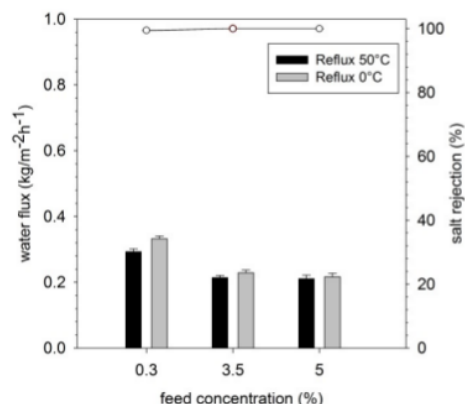


Figure 8. Water fluxes (vertical bars) and salt rejection values (point) of membranes for temperature reflux (0°C and 50°C) as a function of feed salt concentrations (0.3, 3.5 and 5 wt.%)

Based on Fig 8 is show water flux and salt rejection for membrane calcined at 250°C with reflux temperature of sol gel at 0°C and 50°C as a function of feed salt concentration (0.3, 3.5 and 5% NaCl) at a temperature feed 25°C . The highest performances were shown at 50°C of reflux temperature for all variance feed concentration of 0.3 wt% ($0.3324 \text{ kg.m}^{-2}.\text{h}^{-1}$), 3.5 wt% ($0.2290 \text{ kg.m}^{-2}.\text{h}^{-1}$) and 5 wt% ($0.2168 \text{ kg.m}^{-2}.\text{h}^{-1}$). That shows at reflux 50°C with higher siloxane and Si-C bonds is proven to applicate in desalination and endure while the contact to water ions. Beside silica membranes using two-step sol-gel method using nitric acid and ammonia as co-catalysts delivered high water fluxes at $9.5 \text{ kg m}^{-2}.\text{h}^{-1}$ and $6.8 \text{ kg m}^{-2}.\text{h}^{-1}$ for the 0.3 and 3.5 wt % saline solutions, the water flux is higher this is caused by the pH of solution is 6 disposed to based, so the pore is larger than just only using weak acid catalyst [2]. According to a previous study [10] increasing of feed salt concentration caused decreasing to the water flux. This is related to polarization effects. The concentration of water on the membrane surface decreases when water infiltrate through the membrane, resulting in increased salt concentration on the membrane surface. Hence, the polarization effect of salt will tend to limit driving force to water permeation. Furthermore, decreasing of water flux value is also caused by pore blockage in the membrane. Salt ions would blocked pore because of the high salt concentration [11].

In this study, the high rejection salts obtained exceeded 99% for all concentrations, this result is explained by the very low salt flux through the membrane. The high salt rejection is caused by the pore size of membrane is the mesopore which salt ion particles which have a larger size than the membrane pore, so that cannot pass through the membrane easily. Compared to previous research by Wijaya, Duke and Diniz da Costa [12] the highest water flux can be obtained in 3.5 wt% NaCl concentration of membrane performance is $1.7 \text{ kg.m}^{-2}.\text{h}^{-1}$. The flux was higher than this work. However, percentage of salt rejection was lower, that is only 77%. Then compared to Lin, Ding, Smart and Diniz da Costa [13], the water flux in NaCl concentration of 1 wt% also higher and the

salt rejection was similar with this work of $1.8 \text{ kg.m}^{-2}.\text{h}^{-1}$ and 99.5%, respectively. Nevertheless, the pervaporation was operated at high temperature of 75°C . These comparisons shown the membrane that fabricated in this work also has a good structures and performances that promising to use further in water desalination.

Conclusion

The mesoporous silica materials have been employed successfully. Sol gels was chosen as the method in synthesise mesoporous silica. The single organo catalyst using in this work give the good influence to the network of silica matrices in this materials. The FTIR was show the best result was obtained at reflux 50°C with high content of siloxane and Si-C bonds. The best surface area and total pore volume were obtained at 0°C with slightly higher than 50°C ($354 \text{ m}^2/\text{g}$ for surface area and $0.215 \text{ cm}^3/\text{g}$). All sample were categorized as mesoporous materials. Good performance in desalination application was obtained at membrane that calcined at 250°C with sol gel reflux temperature at 50°C , the water fluxes are $0.3324 \text{ kg.m}^{-2}.\text{h}^{-1}$ (0,3 w.t%), $0.2290 \text{ kg.m}^{-2}.\text{h}^{-1}$ (3.5 w.t%) and $0.2168 \text{ kg.m}^{-2}.\text{h}^{-1}$ (5 w.t%), all membranes gives the excellent salt rejection that is $>99\%$.

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