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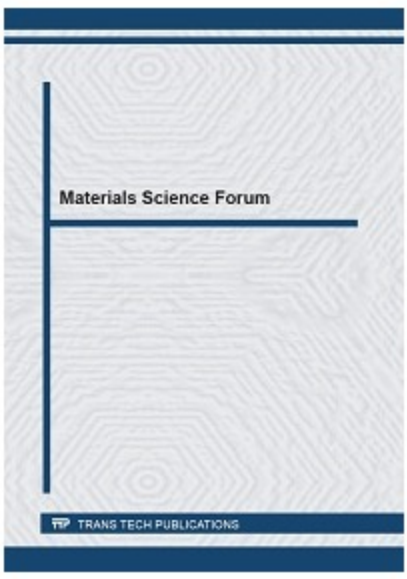

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The Performance of Membranes Interlayer-Free Silica-Pectin Templated for Seawater Desalination via Pervaporation Operated at High Temperature of Feed Solution

Abstract:

Recently, water scarcity is the big issues around the world. Especially in coastal area where the water distribution could not entranced and able to supply clean water for the citizen. The one and only solution is processing seawater to produce fresh and potable water. The desalination process using membrane was recommended to solve this issue. Due to that, the membrane with good structure and high hydro-stability was necessary to fabricate. The aim of this work is to investigate the performance of silica-pectin membranes for treating seawater by pervaporation employing silica based membranes. In this work, the silica-pectin membranes were successfully fabricated using Tetraethyl orthosilicate (TEOS) as silica precursor. Then, pectin from apple was also using in various concentrations (0; 0.1 to 0.5%). This organic material was implemented as a templating agent to produce in silica-pectin thin film. This thin films were dipcoated onto membranes support during membranes fabrication. These membranes were calcined in air at 300 and 400°C using rapid thermal processing (RTP) technique. All membranes were tested for water desalination via pervaporation set-up in various feed temperatures (25, 40 and 60°C). Results show that the membranes produced were crack-free and no pore dense. The FTIR-spectra and Fityk analysis refer to membrane of 2.5% at 300°C and 0.5% at 400°C are the optimum condition due to silanol and siloxane concentrations. An excellent performance was obtained at 0.5% at 400°C with water flux of 8.3 $kg.m^{-2}.h^{-1}$ and high salt rejection of 99.4% at 60 °C of feed temperature. It clearly demonstrates that the silica-pectin membrane has a robust structures due to the templating of carbon chains into silica matrices. The presence of carbon chains in silica matrices may form the smaller and robust pores as expected, that makes the excellent salt rejection in high feed temperature.

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The Performance of Membranes Interlayer-Free Silica-Pectin Templated for Seawater Desalination via Pervaporation Operated at High Temperature of Feed Solution

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Keywords: Membrane interlayer-free silica-carbon templated, seawater desalination, rapid thermal processing

Abstract. Recently, water scarcity is the big issues around the world. Especially in coastal area where the water distribution could not entranced and able to supply clean water for the citizen. The one and only solution is processing seawater to produce fresh and potable water. The desalination process using membrane was recommended to solve this issue. Due to that, the membrane with good structure and high hydro-stability was necessary to fabricate. The aim of this work is to investigate the performance of silica-pectin membranes for treating seawater by pervaporation employing silica based membranes. In this work, the silica-pectin membranes were successfully fabricated using Tetraethyl orthosilicate (TEOS) as silica precursor. Then, pectin from apple was also using in various concentrations (0; 0.1 to 0.5%). This organic material was implemented as a templating agent to produce in silica-pectin thin film. This thin films were dipcoated onto membranes support during membranes fabrication. These membranes were calcined in air at 300 and 400°C using rapid thermal processing (RTP) technique. All membranes were tested for water desalination via pervaporation set-up in various feed temperatures (25, 40 and 60°C). Results show that the membranes produced were crack-free and no pore dense. The FTIR-spectra and Fityk analysis refer to membrane of 2.5% at 300°C and 0.5% at 400°C are the optimum condition due to silanol and siloxane concentrations. An excellent performance was obtained at 0.5% at 400°C with water flux of 8.3 $kg.m^{-2}.h^{-1}$ and high salt rejection of 99.4% at 60 °C of feed temperature. It clearly demonstrates that the silica-pectin membrane has a robust structures due to the templating of carbon chains into silica matrices. The presence of carbon chains in silica matrices may form the smaller and robust pores as expected, that makes the excellent salt rejection in high feed temperature.

Introduction

The water scarcity is the big issues in the world, including Indonesia. The clean water crisis has occurred mainly in coastal areas because of the inaccessibility of water service, causing coastal residents in Indonesia to use well water (groundwater). However, the ground water cannot guarantee the water consumptions of residents for future. Moreover, it is due to the salinity of water is above the standard maximum [1] of drinking water. Water desalination of saline water including brackish water, seawater and brine water is effectively considered to solve the issue of water crisis in coastal areas [2].

The silica membrane has a good thermal and mechanical stability that is suitable to appertain for water desalination. Literally, the synthesized silica membrane has a mesoporous between 3-5 Å

where the water molecule (dk = 2.5 Å) can pass through it well and sieve the salt molecule (i.e. Na +: dk = 7.2 Å and Cl-: dk = 6.6 Å) [3, 4]. The modification of this synthesized silica membrane to form the mesoporous structure was carried out using dual catalysed acid-base [5]. Unfortunately, the long term membrane performance of this structure is not working perfectly. It is because the contact between pores and water molecules during long period makes the pores easily collapsed [5, 6]. It also indicates the lower hydro-phobicity of the membrane itself. From previous work, Elma, Wang, Yacou, Motuzas and Costa [4], Elma, Wang, ChristelleYacou and Costa [6] has been successfully synthesized the interlayer-free silica membranes using cobalt oxide and carbon templates from Pluronic P123. In this work, the silica matrices have been modified by templating carbon chains into the silica matrices using organic material (pectin extracted from apple). This first application of using pectin in membrane fabrication applied for water desalination. Whereas if the membrane is only from pectin without added silica, it may causes the membrane performance easily decline. Then the latest hybrid silica-pectin membrane synthesized by Rangelova, Aleksandrov and Nenkova [7] using a multi-step sol-gel (acid catalyst) method with a combination of TEOS and pectin from apples. Unfortunately, this work has not been applied in the desalination process.

Therefore, in this study the development was carried out by synthesizing interlayer-free silica membranes with pectin as carbon templates using dual acid-base catalyst. The effect of calcination temperatures during membranes fabrication is also investigated in producing mesoporous silica based membranes [3, 8].

Methodology

Xerogel synthesis and characterization. Silica sol were synthesized like our previous work [9]. The Final pH of sol was adjusted to pH 6 ± 0.1. In the meantime, pectin used for carbon templated was prepared by diluting the extracted pectin powder into the glycerol at 40°C for 45 minutes. Finally, the mixture of pectin-glycerol (0.1 to 0.5 wt %) was added into silica mixtures to form silica-pectin sols. The final molar ratios of this sols is the TEOS:EtOH:H_2O:NH_3:HNO_3:pectin sol were calculated to be 1:38:5:0.00008:0.0003:x, where x was variation of pectin concentrations from 0.000013 (0.1 %) to 0.00035 (2.5 %). After that, the silica-pectin sol was dried into oven for 24 h then grounded as xerogels for characterization. The powder of silica-pectin xerogels were calcined in air at 300°C and 400°C for 1 hour without applying ramping rate and dwelling time (RTP: Rapid Thermal Processing Technique).

Membrane preparation and characterization. Silica-pectin thin film were coated directly onto macroporous alumina substrates like previous work [10-12]. Then the coated membrane was dried into an oven and calcined in air using a furnace for 1 h at 300 and 400°C using RTP technique.

The Fourier Transform Infra-Red (FTIR) spectra was carried out to measure the functional groups in silica sol. Peaks were deconvoluted using Fityk software for over the region 1300-700 cm^{-1} with an approximately 5% of errors. The morphology and thickness of membranes were characterized by SEM (Scanning Electron Microscopy), SEM ZEISS EVO® LS15.

Desalination application. The membrane was installed in pervaporation set-up for desalination. One of the membrane side was blocked (dead end mode) and connected to vacuum line with absolute pressure 1 bar. For other side was immersed in the reactor containing feed solution (NaCl, 3.5 wt%, Sigma-Aldrich) at various temperatures of 25°C to 60°C ± 2°C. The feed solution were stirred to avoid concentration polarisation. The water flux, F (kg m^{-2} h^{-1}), was calculated based on equation $F = m/(A\Delta t)$, where m is permeate mass (kg) retained in the cold trap, A is surface-active area of membrane (m^2) and Δt is operation time (h). The salt rejection, R (%), was determined based on equation $R = (C_f - C_p)/C_f \times 100\%$, where C_f and C_p are the feed and permeate concentrations were correlated to conductivities of the retentate and permeate solutions determined by a conductivity metre (OHAUS SF300C-G).

Results and Discussion

Xerogel characterization. The FTIR spectra data for all xerogel samples were shown at **Fig. 1.** The result shows a similar vibrational bands in region 1400-600 cm^{-1}. The intense peak of 1061, 1078, 1173 and 1180 cm^{-1} are indicates the presence of siloxane (groups). Meanwhile, the silanol groups were detected at wavelength 959 and 960 cm^{-1}. The other peak was shown at the FTIR graph and indicates the silica-carbon chains at wavelength ~800 cm^{-1}. All samples show the silica-pectin chains with different high of the peaks. It is clearly shown that the calcination temperatures influence the polymerisation of carbon group templates into silica matrices. The silica-pectin chains exist because of the carbon templated from pectin apples. The same result also reported by previous research of [13]. The carbon adding into silica matrices can contributed to makes the strengthen network and enhancing the hidro-stability of membrane [3, 6, 13, 14].

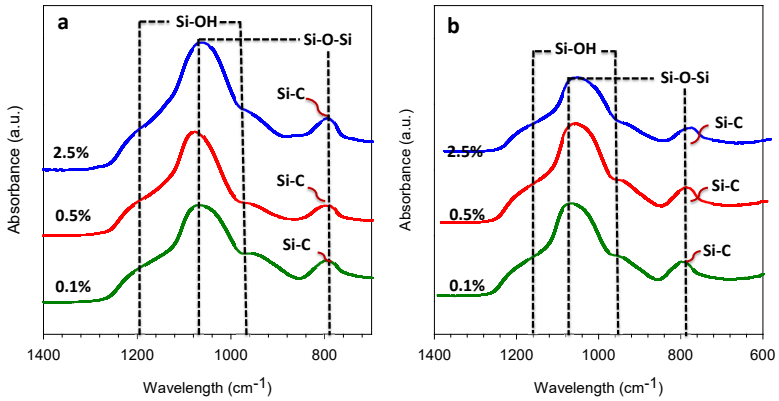

Fig. 1. FTIR Spectra of Xerogel Calcined at 300°C (a) and 400°C (b) via RTP Technique with Various Pectin Concentrations

The highest peak of the siloxane peak of silica-pectin xerogel of 2.5% calcined at 300°C. However, in order to ensure the siloxane and silanol concentration, the amount of areas were evaluated using Fityk software [5]. The peak area ratios of silanol against siloxane groups was analysed and presented in **Fig. 2.** The lowest peak area ratio was shown at xerogel samples of 0.5 and 2.5 wt% calcined in air at 400 and 300°C, respectively. The results observed the surface areas of siloxane is much higher than silanol groups areas. High concentration of siloxane groups represent the mesoporous or microporous [5, 6] in silica-pectin materials. Conversely, the highest silanol group contributed to formed microporous.

Literally, the silica-carbon chains in silica membranes matrices also contribute to the pores. The carbon chains tended to formed small pores, however, it may strengthen Si-OH. Elma, Wang, Yacou and Costa [15] reported that the bonding of carbon chains such as silica-carbon create a good and stronger silica network of silica based membranes that may impact to the mechanical and thermal stability of membrane performances. This result show similar data saying that the carbon chains produced from pectin (apples) strengthen the hydrostability of the materials. Rangelova, Aleksandrov and Nenkova [7] reported that high bonding of carbons in material gives the higher hydrostability. However, the temperature of calcination may influence the carbon chains bonding in the silica-pectin materials. It is due to carbon is an organic material and easy to burn.

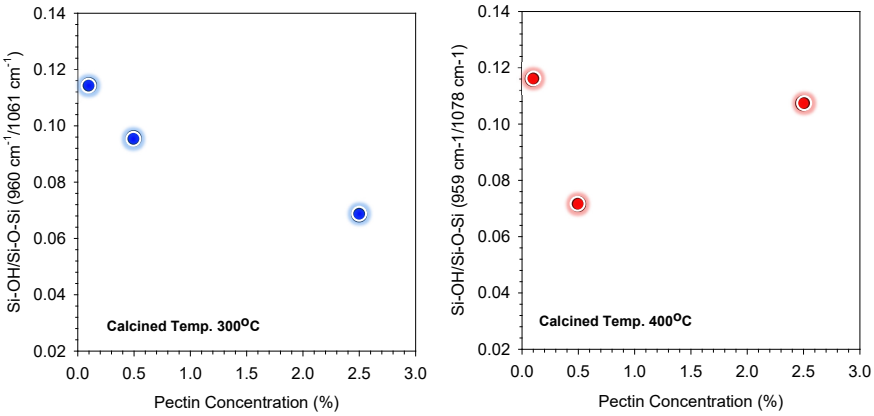

Fig. 2. Peak Area Ratio Si-OH Against Si-O-Si for Xerogel Samples in Various Pectin Concentrations and Calcined at (a) 300 and (b) 400 °C

Membrane morphology. The result of silica-pectin membrane morphology analysis presented in SEM images that is shown in **Fig. 3**. The top surface (**Fig. 3 (a)**) of the silica-pectin membrane of 0.5% calcined at 400°C shows the crack-free and found there is no defect on the membranes. Through the image, the membrane surfaces were not smooth, it is due to the interlayer-free of membranes during fabrication processes. The ramping and cooling rates were also not applied during membrane fabrication, where it is affected by the application of RTP technique. These similar conditions were also found of work from previous researcher [3, 16, 17]. Then the **Fig. 3 (b)** shows the cross-section image of the membrane. The thickness of silica-pectin membrane was measured approximately of 1.5 µm that thicker than our previous research (~0.5 µm). It is also caused by different calcination techniques (RTP technique). This RTP technique was only implemented for 1 h which is basically faster than CTP (Conventional Thermal Processing) technique, 4 h per layer [4]. The perfect evaporation of solvent and water were conducted to the time of the process as the function [18].

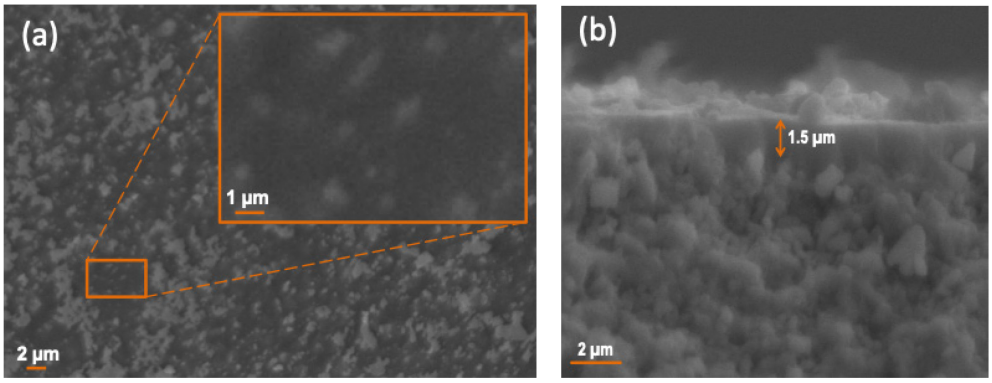

Fig. 3. SEM Images of Silica-pectin Membrane of 0.5% (a) Top Surface and (b) Cross-section Calcined in Air at 400°C

Interlayer-free of membranes here means it is or not applying interlayer materials in membranes. In addition, this work also contributed to the thickness of thin film and the pores. The silica-pectin sols directly infiltrated into the pores of membrane support (macroporous alumina γ-Al_2O_3). This modification technique reduce the time and cost during membrane fabrication. From previous works were also reported that no interlayer and RTP techniques are an excellent strategy in membrane fabrication that also give a good performance.

Membrane performance. Membrane performance was measured and investigated in seawater (3.2 wt%) at varied feed temperature 25 to 60°C ± 2°C as shown in **Fig. 4**. The result shows the water flux increases by feed temperature but in contrary with the salt rejection. The higher

temperature of feed solution affects to the increasing of the vapor pressure as the driving force of permeating processes [15]. That makes a high rate of evaporation and also the steam were then converted to water or permeate. On the other hand, higher feed temperature affect to the decreasing of the salt rejection. It could be due to the membranes pores are not strong enough when the pervaporation processed at high feed temperature [6]. The highest performance was obtained in silica-pectin membrane of 2.5 wt% calcined at 300°C (8.94 $kg.m^{-2}.h^{-1}$) and 0,5 wt% calcined at 400°C (8.62 $kg.m^{-2}.h^{-1}$) for feed temperature of 60°C.

It also observed from the salt rejection values of membranes of the 0.5 wt% of silica-pectin membrane calcined at 400°C is the optimum condition with excellent salt rejection of 99.4%. This phenomena could be explained that there is an influence of calcination temperature to silica-carbon chains in membranes matrices. Because carbon is an organic material and easy to burn at high temperature. Although the performance of silica-pectin membrane of 2.5 wt% is higher than 0.5 wt%, however, the calcination temperature for silica-pectin membrane of 0.5% is appropriate that may strengthen pores better than silica-pectin membrane of 2.5 wt%.

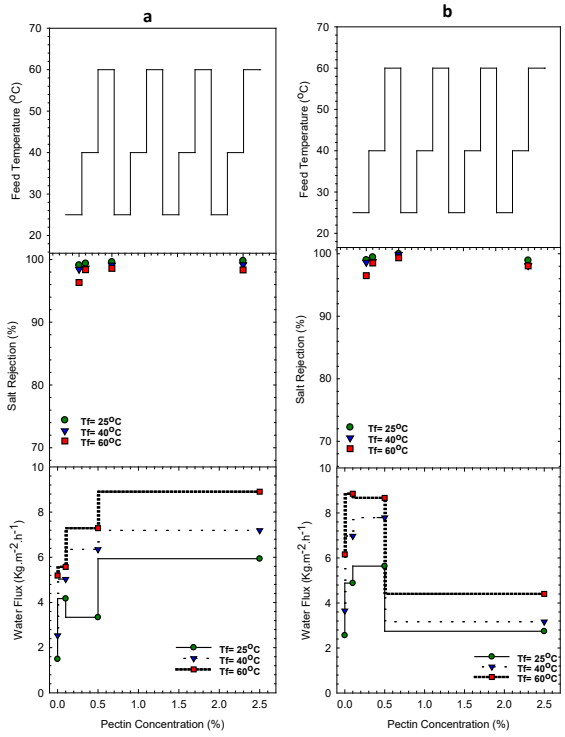

Fig. 4. The Water Fluxes and Salt Rejection Values of Silica-pectin Membrane Calcined at (a) 300 and (b) 400 with function of Pectin Concentration (0; 0.1; 0.5; and 2.5%) and with Feed Temperatures (25, 40 and 60°C)

When these results were compared to other work of inorganic membranes from some literatures (**Table 1**), It is clearly found that the desalination performance is better than other work. However, if comparing to other work from Elma, Yacou, Costa and Wang [5] about the pure silica interlayer-free membrane by CTP technique, this work shows slightly better performance compare to this work. It caused by CTP technique applied during membrane calcination process. In other word, RTP technique applied for this work is more efficient than CTP technique it takes only 1 h for the calcination process in every membrane layer which is different than work done by Elma, Yacou, Costa and Wang [5]. It can be concluded that calcination technique influence in producing the robust structures of silica network. Another phenomena that happened in this water desalination is attributed to the ability of hydrophilic silica surface to adsorb hydrated salt ions as reported by [16]. The water is not only evaporated, but also it has an interaction with the hydrophilic silica occurred.

Table 1. Comparison of Silica Membranes Performances in Seawater Desalination

Membrane	Calcination Method	Feed Temperature (°C)	Water Flux ($kg.m^{-2}.h^{-1}$)	Salt Rejection (%)	Ref.
Pure silica membrane	CTP (air)	25	6.8	98.2	[5]
Silica-cobalt membrane (Co-35)	CTP (air)	25	~ 5	>99	[4]
P123 carbonized template silica membrane	CTP (vacuum)	25	~ 2.5	>99	[6]
Interlayer-free membrane ES-40	RTP (air) 630 °C	60	17.8	>99	[16]
Interlayer-free Silica-pectin Membrane 2,5 %-Tc 300 °C	**RTP (in air)**	**25** **60**	**5.9** **8.9**	**99.8** **98.3**	**This work**
Interlayer-free Silica-pectin Membrane 0,5 %-Tc 400 °C	**RTP (in air)**	**25** **60**	**5.6** **8.66**	**99.9** **99.3**	**This work**

Conclusion

The silica-pectin membrane was successfully fabricated with sol-gel method using RTP as calcination techniques. The membrane synthesized in this work shows high siloxane concentration with silica-carbon chains. An optimum membrane was found for membranes templated with pectin 0.5 wt% calcined in temperature 400°C. The morphology of membrane shows no defect, crack-free. The thickness of the membrane (~1.5 μm) is higher than fabricated using CTP techniques. An excellent membranes performance was investigated and show high water fluxes (8.3 $kg.m^{-2}.h^{-1}$) and salt rejection (99.4 %). The membranes work well up to 60°C of feed temperature. The carbon chains from pectin apple is very contributed to the strengthen structure of membrane pores. The silica-pectin membranes is very promising to development for future research and applied for water desalination.

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The Performance of Membranes Interlayer-Free Silica-Pectin Templated for Seawater Desalination via Pervaporation Operated at High Temperature of Feed Solution

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Abstract. Recently, water scarcity is the big issues around the world. Especially in coastal area where the water distribution could not entranced and able to supply clean water for the citizen. The one and only solution is processing seawater to produce fresh and potable water. The desalination process using membrane was recommended to solve this issue. Due to that, the membrane with good structure and high hydro-stability was necessary to fabricate. The aim of this work is to investigate the performance of silica-pectin membranes for treating seawater by pervaporation employing silica based membranes. In this work, the silica-pectin membranes were successfully fabricated using Tetraethyl orthosilicate (TEOS) as silica precursor. Then, pectin from apple was also using in various concentrations (0; 0.1 to 0.5%). This organic material was implemented as a templating agent to produce in silica-pectin thin film. This thin films were dipcoated onto membranes support during membranes fabrication. These membranes were calcined in air at 300 and 400°C using rapid thermal processing (RTP) technique. All membranes were tested for water desalination via pervaporation set-up in various feed temperatures (25, 40 and 60°C). Results show that the membranes produced were crack-free and no pore dense. The FTIR-spectra and Fityk analysis refer to membrane of 2.5% at 300°C and 0.5% at 400°C are the optimum condition due to silanol and siloxane concentrations. An excellent performance was obtained at 0.5% at 400°C with water flux of 8.3 $kg.m^{-2}.h^{-1}$ and high salt rejection of 99.4% at 60 °C of feed temperature. It clearly demonstrates that the silica-pectin membrane has a robust structures due to the templating of carbon chains into silica matrices. The presence of carbon chains in silica matrices may form the smaller and robust pores as expected, that makes the excellent salt rejection in high feed temperature.

Introduction

The water scarcity is the big issues in the world, including Indonesia. The clean water crisis has occurred mainly in coastal areas because of the inaccessibility of water service, causing coastal residents in Indonesia to use well water (groundwater). However, the ground water cannot guarantee the water consumptions of residents for future. Moreover, it is due to the salinity of water is above the standard maximum [1] of drinking water. Water desalination of saline water including brackish water, seawater and brine water is effectively considered to solve the issue of water crisis in coastal areas [2].

The silica membrane has a good thermal and mechanical stability that is suitable to appertain for water desalination. Literally, the synthesized silica membrane has a mesoporous between 3-5 Å

where the water molecule (dk = 2.5 Å) can pass through it well and sieve the salt molecule (i.e. Na +: dk = 7.2 Å and Cl-: dk = 6.6 Å) [3, 4]. The modification of this synthesized silica membrane to form the mesoporous structure was carried out using dual catalysed acid-base [5]. Unfortunately, the long term membrane performance of this structure is not working perfectly. It is because the contact between pores and water molecules during long period makes the pores easily collapsed [5, 6]. It also indicates the lower hydro-phobicity of the membrane itself. From previous work, Elma, Wang, Yacou, Motuzas and Costa [4], Elma, Wang, ChristelleYacou and Costa [6] has been successfully synthesized the interlayer-free silica membranes using cobalt oxide and carbon templates from Pluronic P123. In this work, the silica matrices have been modified by templating carbon chains into the silica matrices using organic material (pectin extracted from apple). This first application of using pectin in membrane fabrication applied for water desalination. Whereas if the membrane is only from pectin without added silica, it may causes the membrane performance easily decline. Then the latest hybrid silica-pectin membrane synthesized by Rangelova, Aleksandrov and Nenkova [7] using a multi-step sol-gel (acid catalyst) method with a combination of TEOS and pectin from apples. Unfortunately, this work has not been applied in the desalination process.

Therefore, in this study the development was carried out by synthesizing interlayer-free silica membranes with pectin as carbon templates using dual acid-base catalyst. The effect of calcination temperatures during membranes fabrication is also investigated in producing mesoporous silica based membranes [3, 8].

Methodology

Xerogel synthesis and characterization. Silica sol were synthesized like our previous work [9]. The Final pH of sol was adjusted to pH 6 ± 0.1. In the meantime, pectin used for carbon templated was prepared by diluting the extracted pectin powder into the glycerol at 40°C for 45 minutes. Finally, the mixture of pectin-glycerol (0.1 to 0.5 wt %) was added into silica mixtures to form silica-pectin sols. The final molar ratios of this sols is the TEOS:EtOH:H_2O:NH_3:HNO_3:pectin sol were calculated to be 1:38:5:0.00008:0.0003:x, where x was variation of pectin concentrations from 0.000013 (0.1 %) to 0.00035 (2.5 %). After that, the silica-pectin sol was dried into oven for 24 h then grounded as xerogels for characterization. The powder of silica-pectin xerogels were calcined in air at 300°C and 400°C for 1 hour without applying ramping rate and dwelling time (RTP: Rapid Thermal Processing Technique).

Membrane preparation and characterization. Silica-pectin thin film were coated directly onto macroporous alumina substrates like previous work [10-12]. Then the coated membrane was dried into an oven and calcined in air using a furnace for 1 h at 300 and 400°C using RTP technique.

The Fourier Transform Infra-Red (FTIR) spectra was carried out to measure the functional groups in silica sol. Peaks were deconvoluted using Fityk software for over the region 1300-700 cm^{-1} with an approximately 5% of errors. The morphology and thickness of membranes were characterized by SEM (Scanning Electron Microscopy), SEM ZEISS EVO® LS15.

Desalination application. The membrane was installed in pervaporation set-up for desalination. One of the membrane side was blocked (dead end mode) and connected to vacuum line with absolute pressure 1 bar. For other side was immersed in the reactor containing feed solution (NaCl, 3.5 wt%, Sigma-Aldrich) at various temperatures of 25°C to 60°C ± 2°C. The feed solution were stirred to avoid concentration polarisation. The water flux, F (kg m^{-2} h^{-1}), was calculated based on equation $F = m/(A\Delta t)$, where m is permeate mass (kg) retained in the cold trap, A is surface-active area of membrane (m^2) and Δt is operation time (h). The salt rejection, R (%), was determined based on equation $R = (C_f - C_p)/C_f \times 100\%$, where C_f and C_p are the feed and permeate concentrations were correlated to conductivities of the retentate and permeate solutions determined by a conductivity metre (OHAUS SF300C-G).

Results and Discussion

Xerogel characterization. The FTIR spectra data for all xerogel samples were shown at **Fig. 1.** The result shows a similar vibrational bands in region 1400-600 cm^{-1}. The intense peak of 1061, 1078, 1173 and 1180 cm^{-1} are indicates the presence of siloxane (groups). Meanwhile, the silanol groups were detected at wavelength 959 and 960 cm^{-1}. The other peak was shown at the FTIR graph and indicates the silica-carbon chains at wavelength ~800 cm^{-1}. All samples show the silica-pectin chains with different high of the peaks. It is clearly shown that the calcination temperatures influence the polymerisation of carbon group templates into silica matrices. The silica-pectin chains exist because of the carbon templated from pectin apples. The same result also reported by previous research of [13]. The carbon adding into silica matrices can contributed to makes the strengthen network and enhancing the hidro-stability of membrane [3, 6, 13, 14].

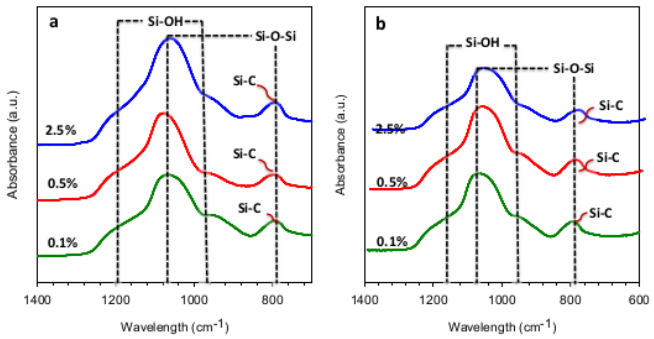

Fig. 1. FTIR Spectra of Xerogel Calcined at 300°C (a) and 400°C (b) via RTP Technique with Various Pectin Concentrations

The highest peak of the siloxane peak of silica-pectin xerogel of 2.5% calcined at 300°C. However, in order to ensure the siloxane and silanol concentration, the amount of areas were evaluated using Fityk software [5]. The peak area ratios of silanol against siloxane groups was analysed and presented in **Fig. 2.** The lowest peak area ratio was shown at xerogel samples of 0.5 and 2.5 wt% calcined in air at 400 and 300°C, respectively. The results observed the surface areas of siloxane is much higher than silanol groups areas. High concentration of siloxane groups represent the mesoporous or microporous [5, 6] in silica-pectin materials. Conversely, the highest silanol group contributed to formed microporous.

Literally, the silica-carbon chains in silica membranes matrices also contribute to the pores. The carbon chains tended to formed small pores, however, it may strengthen Si-OH. Elma, Wang, Yacou and Costa [15] reported that the bonding of carbon chains such as silica-carbon create a good and stronger silica network of silica based membranes that may impact to the mechanical and thermal stability of membrane performances. This result show similar data saying that the carbon chains produced from pectin (apples) strengthen the hydrostability of the materials. Rangelova, Aleksandrov and Nenkova [7] reported that high bonding of carbons in material gives the higher hydrostability. However, the temperature of calcination may influence the carbon chains bonding in the silica-pectin materials. It is due to carbon is an organic material and easy to burn.

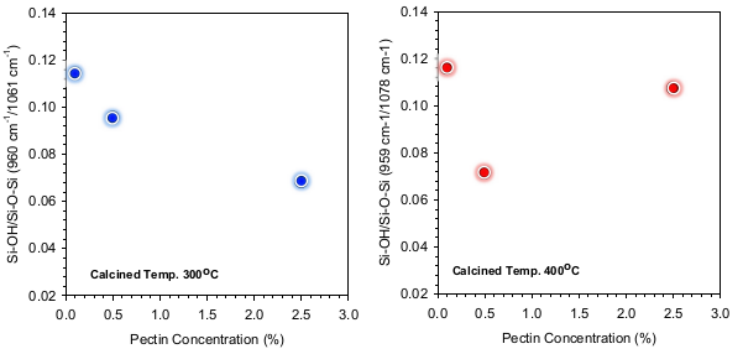

Fig. 2. Peak Area Ratio Si-OH Against Si-O-Si for Xerogel Samples in Various Pectin Concentrations and Calcined at (a) 300 and (b) 400 °C

Membrane morphology. The result of silica-pectin membrane morphology analysis presented in SEM images that is shown in **Fig. 3**. The top surface (**Fig. 3 (a)**) of the silica-pectin membrane of 0.5% calcined at 400°C shows the crack-free and found there is no defect on the membranes. Through the image, the membrane surfaces were not smooth, it is due to the interlayer-free of membranes during fabrication processes. The ramping and cooling rates were also not applied during membrane fabrication, where it is affected by the application of RTP technique. These similar conditions were also found of work from previous researcher [3, 16, 17]. Then the **Fig. 3 (b)** shows the cross-section image of the membrane. The thickness of silica-pectin membrane was measured approximately of 1.5 µm that thicker than our previous research (~0.5 µm). It is also caused by different calcination techniques (RTP technique). This RTP technique was only implemented for 1 h which is basically faster than CTP (Conventional Thermal Processing) technique, 4 h per layer [4]. The perfect evaporation of solvent and water were conducted to the time of the process as the function [18].

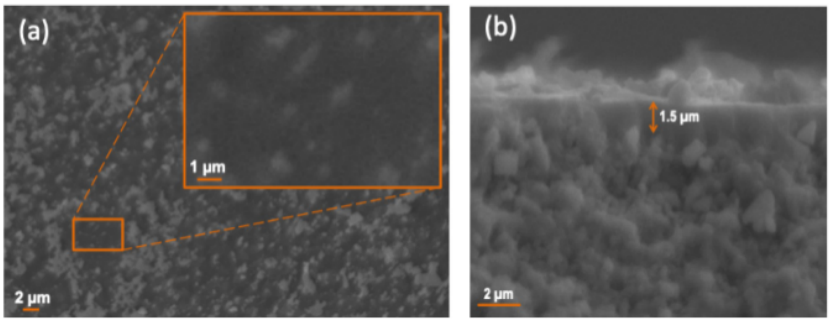

Fig. 3. SEM Images of Silica-pectin Membrane of 0.5% (a) Top Surface and (b) Cross-section Calcined in Air at 400°C

Interlayer-free of membranes here means it is or not applying interlayer materials in membranes. In addition, this work also contributed to the thickness of thin film and the pores. The silica-pectin sols directly infiltrated into the pores of membrane support (macroporous alumina γ-Al_2O_3). This modification technique reduce the time and cost during membrane fabrication. From previous works were also reported that no interlayer and RTP techniques are an excellent strategy in membrane fabrication that also give a good performance.

Membrane performance. Membrane performance was measured and investigated in seawater (3.2 wt%) at varied feed temperature 25 to 60°C ± 2°C as shown in **Fig. 4**. The result shows the water flux increases by feed temperature but in contrary with the salt rejection. The higher

temperature of feed solution affects to the increasing of the vapor pressure as the driving force of permeating processes [15]. That makes a high rate of evaporation and also the steam were then converted to water or permeate. On the other hand, higher feed temperature affect to the decreasing of the salt rejection. It could be due to the membranes pores are not strong enough when the pervaporation processed at high feed temperature [6]. The highest performance was obtained in silica-pectin membrane of 2.5 wt% calcined at 300°C (8.94 $kg.m^{-2}.h^{-1}$) and 0,5 wt% calcined at 400°C (8.62 $kg.m^{-2}.h^{-1}$) for feed temperature of 60°C.

It also observed from the salt rejection values of membranes of the 0.5 wt% of silica-pectin membrane calcined at 400°C is the optimum condition with excellent salt rejection of 99.4%. This phenomena could be explained that there is an influence of calcination temperature to silica-carbon chains in membranes matrices. Because carbon is an organic material and easy to burn at high temperature. Although the performance of silica-pectin membrane of 2.5 wt% is higher than 0.5 wt%, however, the calcination temperature for silica-pectin membrane of 0.5% is appropriate that may strengthen pores better than silica-pectin membrane of 2.5 wt%.

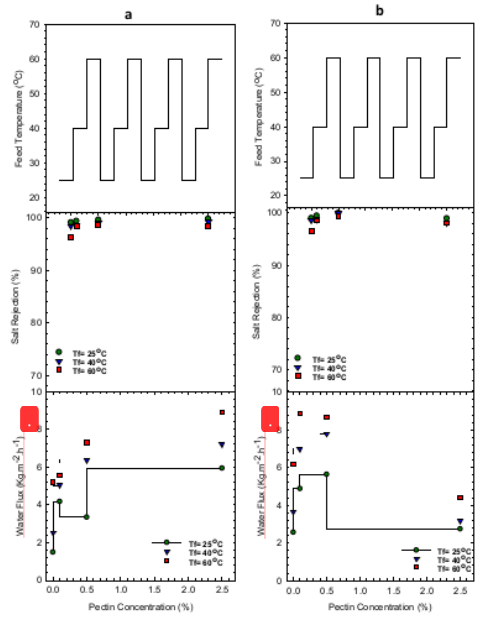

Fig. 4. The Water Fluxes and Salt Rejection Values of Silica-pectin Membrane Calcined at (a) 300 and (b) 400 with function of Pectin Concentration (0; 0.1; 0.5; and 2.5%) and with Feed Temperatures (25, 40 and 60°C)

When these results were compared to other work of inorganic membranes from some literatures (**Table 1**), It is clearly found that the desalination performance is better than other work. However, if comparing to other work from Elma, Yacou, Costa and Wang [5] about the pure silica interlayer-free membrane by CTP technique, this work shows slightly better performance compare to this work. It caused by CTP technique applied during membrane calcination process. In other word, RTP technique applied for this work is more efficient than CTP technique it takes only 1 h for the calcination process in every membrane layer which is different than work done by Elma, Yacou, Costa and Wang [5]. It can be concluded that calcination technique influence in producing the robust structures of silica network. Another phenomena that happened in this water desalination is attributed to the ability of hydrophilic silica surface to adsorb hydrated salt ions as reported by [16]. The water is not only evaporated, but also it has an interaction with the hydrophilic silica occurred.

Table 1. Comparison of Silica Membranes Performances in Seawater Desalination

Membrane	Calcination Method	Feed Temperature (°C)	Water Flux ($kg.m^{-2}.h^{-1}$)	Salt Rejection (%)	Ref.
Pure silica membrane	CTP (air)	25	6.8	98.2	[5]
Silica-cobalt membrane (Co-35)	CTP (air)	25	~ 5	>99	[4]
P123 carbonized template silica membrane	CTP (vacuum)	25	~ 2.5	>99	[6]
Interlayer-free membrane ES-40	RTP (air) 630 °C	60	17.8	>99	[16]
Interlayer-free Silica-pectin Membrane 2,5 %-Tc 300 °C	**RTP (in air)**	**25** **60**	**5.9** **8.9**	**99.8** **98.3**	**This work**
Interlayer-free Silica-pectin Membrane 0,5 %-Tc 400 °C	**RTP (in air)**	**25** **60**	**5.6** **8.66**	**99.9** **99.3**	**This work**

Conclusion

The silica-pectin membrane was successfully fabricated with sol-gel method using RTP as calcination techniques. The membrane synthesized in this work shows high siloxane concentration with silica-carbon chains. An optimum membrane was found for membranes templated with pectin 0.5 wt% calcined in temperature 400°C. The morphology of membrane shows no defect, crack-free. The thickness of the membrane (~1.5 µm) is higher than fabricated using CTP techniques. An excellent membranes performance was investigated and show high water fluxes (8.3 $kg.m^{-2}.h^{-1}$) and salt rejection (99.4 %). The membranes work well up to 60°C of feed temperature. The carbon chains from pectin apple is very contributed to the strengthen structure of membrane pores. The silica-pectin membranes is very promising to development for future research and applied for water desalination.

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