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Performance of Cobalt-Silica Membranes through Pervaporation Process with Different Feed Solution Concentrations

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Abstract:

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Microscopy (SEM) and Brunauer-Emmett-Teller (BET). From the FTIR result, it is known that the higher the concentration of cobalt in the membrane, the more silanol and siloxane groups. Based on BET analysis, Si-Co 35 wt% membrane has largest pore volume ($0.129387 \text{ cm}^3\text{g}^{-1}$). In addition, the highest value of water flux ($7.2660 \text{ kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$) and salt rejection (100%) is achieved by Si-Co 5% membrane in 0.3% NaCl feed at 60 °C. The value of water flux decreases and the value of salt rejection increases with increasing feed concentration.

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Performance of Cobalt-Silica Membranes Through Pervaporation Process with Different Feed Solution Concentrations

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Keywords: cobalt-silica membranes; pervaporation; water flux; salt rejection.

Abstract. This work shows the performance of cobalt-silica membranes through water desalination via pervaporation process. The aim of this work is to find out the performance of the cobalt oxide as a templating agent in the silica cobalt membranes for water desalination via pervaporation process. It also aims to investigate the water flux and salt rejection of silica cobalt membranes using artificial saline water. The concentration of cobalt oxide as a template for fabricating cobalt-silica membranes were 5 – 35 wt%. The feed solution applied during pervaporation process were 0.3 – 5 wt% NaCl with operating temperatures of 25, 40 and 60 °C. The silica xerogels were characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM) and Brunauer-Emmett-Teller (BET). From the FTIR result, it is known that the higher the concentration of cobalt in the membrane, the more silanol and siloxane groups. Based on BET analysis, Si-Co 35 wt% membrane has largest pore volume ($0.129387 \text{ cm}^3 \text{ g}^{-1}$). In addition, the highest value of water flux ($7.2660 \text{ kg.m}^{-2}.\text{h}^{-1}$) and salt rejection (100%) is achieved by Si-Co 5% membrane in 0.3% NaCl feed at 60 °C. The value of water flux decreases and the value of salt rejection increases with increasing feed concentration.

Introduction

Increasing sea level as a result of climate change causes less fresh water to be used as potable water. Therefore, the source of drinking water from seawater, a process called desalination, is a logical way forward to addressing the water needs of current and future needs. Seawater desalination can be done by membrane separation processes such as reverse osmosis (RO), membrane distillation (MD) or pervaporation (PV).

Membrane technology has long been a separation process in the industrial world because of its fast and efficient process. In general, there are three types of membrane processes that are usually applied, it is called reverse osmosis (RO), membrane distillation (MD) and pervaporation (PV)[1]. Membrane is basically a thin layer between two fluid phases, its feed phase and the permeate phase which is a barrier to a particular species, which can separate substances of different sizes and limit the transport of various species based on physical and chemical properties. Membranes are semipermeable, mean that membrane can hold certain species larger than the membrane pore size and pass other species of smaller size. The selective nature of this membrane can be used in the separation process. Membrane support is a membrane from ceramic material made through high temperature combustion[2]. The stability of membrane strength depends on the chemical, mechanical and thermal conditions of the membrane[3]. Membranes in their application for water treatment do not require the addition of chemicals (coagulants, flocculants) so that they are friendly to the environment[4].

Inorganic membranes have recently caught the attention of the research and industry communities because of their long life span and better resistance to environmental factors and extrinsic industries. Now, there are two types of inorganic silica membranes which have been being

widely observed for water desalination, they are zeolite and amorphous silica membranes. Zeolite membranes have proven effective but long-term stability and improvement still present problems to date. Silica membranes have excellent molecular filtration properties and simple fabrication processes through sol-gel processing, although they need functionalization to overcome hydro-instability. The structure of the silica membrane has a pore size in the range of 3-5 Å while the kinetic diameter of water molecules is 2.6 Å, making it ideal for inhibiting the passage of hydrated salt ions (Na^+ : $dk = 7.2$ Å and Cl^- : $dk = 6.6$ Å). Therefore, the silica membrane separates water from salt with a molecular filtration mechanism.

Methodology

Thin Film Fabrication and Pervaporation. Silica sols were synthesized from a two-step sol gel process employing acid and base as catalysts (followed our previous work [5]). Tetraethyl orthosilicate (TEOS) was added drop-wise into ethanol (EtOH) solution and stirred for 5 minutes in ice bath condition at 0 °C to avoid partial hydrolysis followed by the addition of diluted nitric acid (0.0008 M HNO_3). The sol mixture was refluxed for 1 hours at 50 °C with stirring to achieve a complete hydrolysis of the alkoxy groups. Ammonia solution (NH_3) diluted in ethanol was added drop wisely into sol mixture to commence the condensation reaction and reflux was continued for another 2 hours to obtain the resultant sol. Silica sols solution was then dried in a temperature controlled (oven) at 60 °C for 24 hours to obtain the dried gel for xerogel characterization. The final molar ratios of the sol (TEOS:EtOH: HNO_3 : H_2O : NH_3) were calculated to be 1:38:0.00078:5:0.003. Then, silica sol mixed with cobalt oxide 0.2873 g for Si-Co 5%, 0.5746 g for Si-Co 10%, 1.1492 g for Si-Co 20% and 2.0111 g for Si-Co 35%. Furthermore, Si-Co sol solution (thin membrane films) were coated on macroporous alumina substrates (α - Al_2O_3 tubular support, pore size ≈ 100 nm) via a dip-coating process with a dwell time of 2 minutes and a dipping and withdrawal rate of 10 and 5 $\text{cm}\cdot\text{min}^{-1}$, respectively. After the deposition of each layer of thin film onto membrane support, it was then dried and calcined in air at 600 °C for 1 hours. This cycle of dip-coating and calcination process was repeated up to four layers. Next step is to prepare a feed solution of 0.3; 3.5; 5% of artificial saline water. Each feed solution was placed into the feed tank. Then connect the cold trap and assemble in the classical pervaporation set-up as shown in figure 1. The dead-end mode of tubular Si-Co membrane was set (one of end of tube was blocked and the other was connected to cold trap and vacuum pump). Membrane was immersed into feed solution and then the pervaporation was started in every 20 minutes. The separated solution will evaporate and flow through pore size of membrane and collected in the cold trap. The evaporated molecules was then condensed in the cold trap by immersing cold trap into liquid nitrogen. The following step is to calculate the water flux and measure the salt rejection using a conductivity meter. Then calculations of the water flux and salt rejection were repeated three times in every measurement. The water flux, F ($\text{kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$), was determined based on the equation

$$F = m/(A \cdot \Delta t) \quad (1)$$

where m is the mass of permeate (kg) retained in the cold trap, A is the surface-active area (m^2) and Δt is the time measurement (h). The salt rejection, R (%), was calculated as

$$R = (C_f - C_p)/C_f \times 100\% \quad (2)$$

where C_f and C_p are the feed and permeate concentrations of salt (wt %)

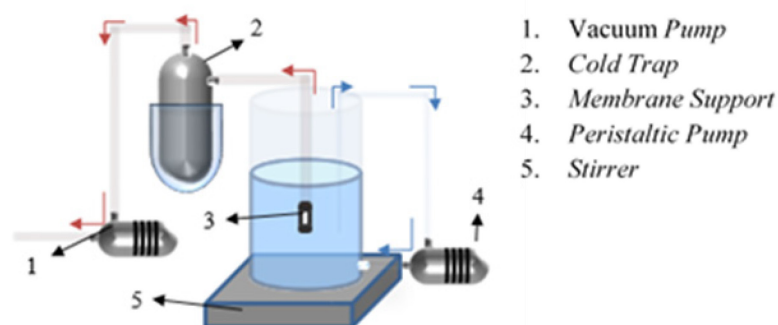


Fig. 1. Desalination rig via classical pervaporation set up

Result and Discussion

Xerogel Characterization. Silica membranes are fabricated from silica synthesis with the sol-gel method that has been developed mainly for gas separation with pore sizes in the range of 3-5 Å[6]. The difficulty of using silica membranes for desalination is related to the instability of the silica matrix when exposed to water[7]. In order to deal with this problem, researchers have implanted and carbonated with covalent templates or surfactants[8] in the silica matrix. Recently, a number of research groups reported the development of metal or metal oxide doping on silica membranes with exceptional gas selectivity, thus showing good pore size reduction. One of the metal oxides is cobalt [9, 10]. In preparing cobalt-silica sol is by applying the sol-gel method, the sol was obtained in this study where soles with a pH of 6 would produce mesoporous membrane pores[5]. It is in order to fabricate the pore size of silica matrices were greater than the pore size of a water molecules. Also, it is smaller than salt molecules. This supposed to water molecules are easily sieved through silica matrices. Literally, silica sol is an aqueous clear colorless liquid, where after adding it to cobalt the color will turn clear purple. In addition to providing strength to the membrane pores, the addition of cobalt in silica sol will affect silanol and siloxane groups. Silanol and siloxane groups are known to affect the membrane pore size, so it is important to know whether or not there are two groups[11]. Using the Fourier Transform Infrared (FTIR) analysis method, it can be seen the area of silanol and siloxane obtained in cobalt-silica sol. From the results of the FTIR analysis the graph is shown in Figure 2(a).

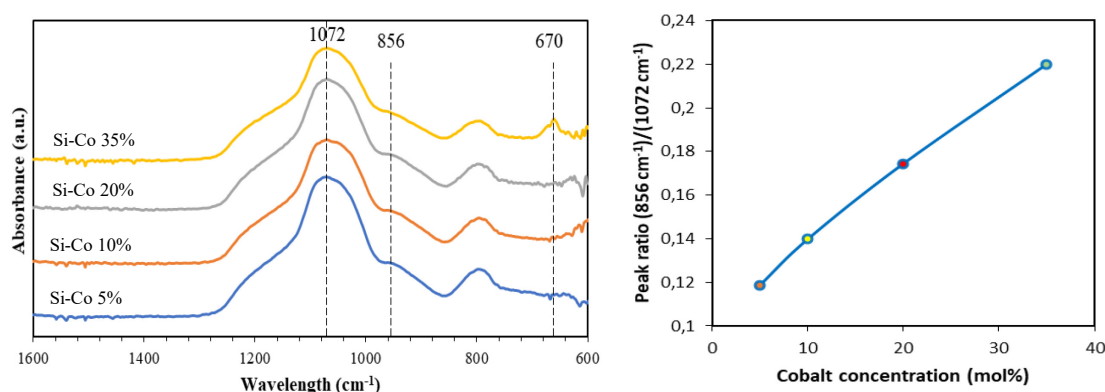


Fig. 2. (a) FTIR spectra of cobalt-silica xerogels, (b) deconvulation data between silanol and siloxane of cobalt-silica xerogels

Figure 2(a) shows the peaks of siloxane groups found in the wavelength 1072 cm^{-1} and 798 cm^{-1} . and the peaks found in the wavelength 856 cm^{-1} is silanol groups. Then, also found Co_3O_4 in the wavelength 607 cm^{-1} . These peaks are slightly similar from previous studies [11]. In figure 2(b) tells about the deconvulation data. Deconvulation data were observed from Fityk software from wavelength 600 to 1600 cm^{-1} . The error of this Fityk software analysis was investigated for about

5%. Gaussian curve was applied to fit the curves. Also, In order to deconvolute peaks, HWHM (the half width half max) has been done for every single peaks. And then, the area of silanol group (856 cm^{-1}) was compared to siloxane groups (1072 cm^{-1}) as well as the concentration of cobalt oxide in the calcined samples (silica xerogels). From the figure 2(b), it can be seen that the deconvoluted area increases with the increasing of the concentration of cobalt in the samples. Moreover, when the concentration of cobalt oxide templated into silica sols increase, so, the silanol groups in the silica matrix also increase while the siloxane groups decrease. This is correlated to the results of Elma's study [11] which also states that the concentration of silanol groups in the silica matrix increases with increasing concentration of cobalt oxide in the silica matrices.

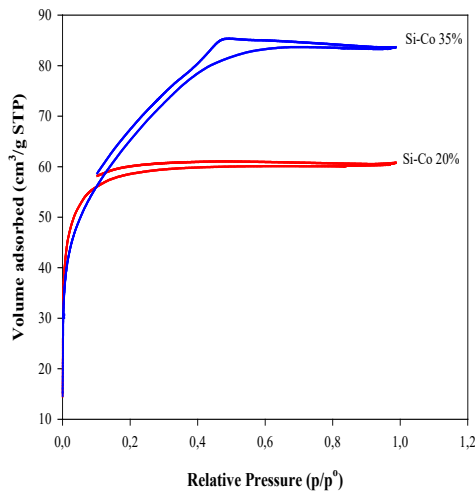


Fig. 3. Plots of N_2 isotherm data of the calcined Co-Si xerogels

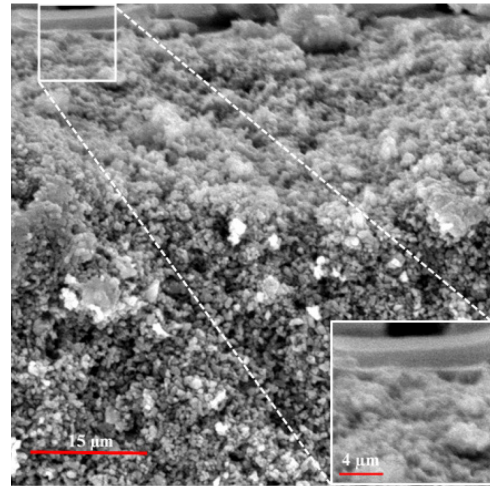


Fig. 4. SEM image cross-section Si-Co 5% membrane

Figure 3 is a graph of isothermal N_2 adsorption on Si-Co xerogels with the concentration of cobalt oxide 20 wt% and Si-Co xerogels with the concentration of cobalt oxide 35 wt%. Based on the results of BET analysis, Si-Co xerogels with the concentration of cobalt oxide 20 wt% has a lower surface area ($188.9547\text{ m}^2\text{g}^{-1}$) than Si-Co xerogels with the concentration of cobalt oxide 35 wt%. Also, it is with a total volume of $0.094015\text{ cm}^3\text{g}^{-1}$. While Si-Co xerogels with the concentration of cobalt oxide 35 wt%. has a high surface area of $225.3542\text{ m}^2\text{g}^{-1}$ with a total volume of $0.129387\text{ cm}^3\text{g}^{-1}$. In addition, the pore size of the Si-Co xerogels with the concentration of cobalt oxide 20 wt% is smaller than the pore size of the Si-Co xerogels with the concentration of cobalt oxide 35 wt%. It is due to the cobalt oxide create the pore size bigger. The higher the content of cobalt oxide in xerogels, the bigger the size of the pores. In a previous study [11] a silica xerogels produces also found in mesoporous size.

Figure 4 is obtained through a SEM (Scanning Electron Microscopic) which shows a cross section of Si-Co membrane 35% which is made through the process of dipcoating and rapid thermal process (RTP). In the first dipcoating process, the thin film then fill the pores of the membrane support which has a big pores ($\sim 100\text{ }\mu\text{m}$), so that the sols will completely meet the membrane pores of support. During the dipcoating process, after the membrane support pores have been filled, then the thin film layer begins to form. And this process will continue up to fourth layer. From figure 4 found that the membrane thickness of Si-Co membrane with the cobalt concentration 35% was $1.9\text{ }\mu\text{m}$. After dipcoating, the membranes were calcined using rapid thermal processing (RTP) technique for 1 h at $600\text{ }^\circ\text{C}$. The calcination process of membranes done after dipcoating process was held in every layer. It is found that silica membranes fabricated through this rapid thermal process (RTP) are thicker than silica membranes which are processed through a conventional calcination processing (CTP) technique. It is because the calcination process using RTP technique is much shorter than (1 h) the membranes fabrication using CTP technique (4 h). It may also cause the solvent from thin film dipcoated onto membrane support is trapped inside the silica matrix during

calcination process, so that the evaporation process during calcination process may be incomplete. The calcination process carried out directly from the room temperature at high temperatures cause the ethanol as a solvent is not completely evaporated. Then, there is still H₂O molecules remained in the silica matrices. This is phenomena is addressed to describe the membranes thickness after fabrication. When compare to the thickness of the membrane fabricated by the conventional calcination processing (CTP) techniques investigated from Elma [11] was 0.4 μm. Interestingly, even though the silica cobalt membranes fabricated from RTP technique is thicker than silica cobalt membranes fabricated from CTP technique, the performance of these membranes is excellent. It then can be seen from the high value of salt rejection in Figure 5.

Water Flux and Salt Rejection. Figure 5 shows the relationship between water flux and salt rejection on various types of silica cobalt membranes with various types of feed solution. As we know that water flux is the ability of a membrane to be passed by water molecules per unit area per unit time. Whereas salt rejection is the ratio between the salt concentration that does not through the membrane pores and the salt concentration initially at the feed. The value of water flux is obtained from the mass of water obtained in the cold trap divided by the surface area of the membrane in contact with the feed and divided by the length of time pervaporation. In 5% Si-Co membrane, the highest water flux is obtained at a 0.3% NaCl feed temperature of 60 °C which is 7.2660 kg.m⁻².h⁻¹ with 100% salt rejection. In 10% Si-Co membrane, the highest water flux is obtained at 10% NaCl feed temperature of 40 °C, which is 4.5383 kg.m⁻².h⁻¹ with salt rejection 88.14%. In the 20% Si-Co membrane, the highest water flux was obtained at a 3.5 wt% NaCl feed temperature of 40 °C which was 3.4951 kg.m⁻².h⁻¹ with salt rejection 84.62 %. In 35 wt% Si-Co membrane, the highest water flux was obtained at a 5% NaCl feed temperature of 60 °C which is 6.6842 kg.m⁻².h⁻¹. From figure 5 also can be seen that water flux decreases with the increasing of feed concentration. In addition, silica cobalt membranes with a high concentration of cobalt also give low water flux compared to water flux in membranes with lower concentrations of cobalt as a templating agent. The highest water flux value of all silica cobalt membranes found in 0.3 wt% NaCl feed. This is due to small amount of salt particles that clog the pores of the membrane itself. So that the value of the water flux obtained become high. The decrease of water flux is caused by the increasing number of salt particles in the feed solution. It is caused by the pores of the membrane become more clogged. While the decrease in water flux to increase the concentration of cobalt in the membrane due to the increasing number of silanol groups along with the increasing concentration of cobalt in the membrane, so more salt particles of water molecules are trapped inside silica matrices. The silanol group is the bond between silica and the -OH group, which cause the silanol pore size to be smaller than the siloxane pore size. -OH which comes into contact with water molecules (H₂O) become more active and give effect to the membrane performance become collapse. The ruins widen the pore at the top but get narrower at the bottom due to the clogged debris. The pore forms becomes an inverted cone shape. It also causes the value of water flux obtained in this study to be lower than that of water flux obtained by Elma [11] due to more concentration of silanol groups in the thin film. The value of salt rejection on each membrane tends to decrease with increasing salt concentration in the feed. Many salt particles clog the pore, with the high thrust the particles are increasingly trying to penetrate the pore, causing damage to the pore and salt particles to escape.

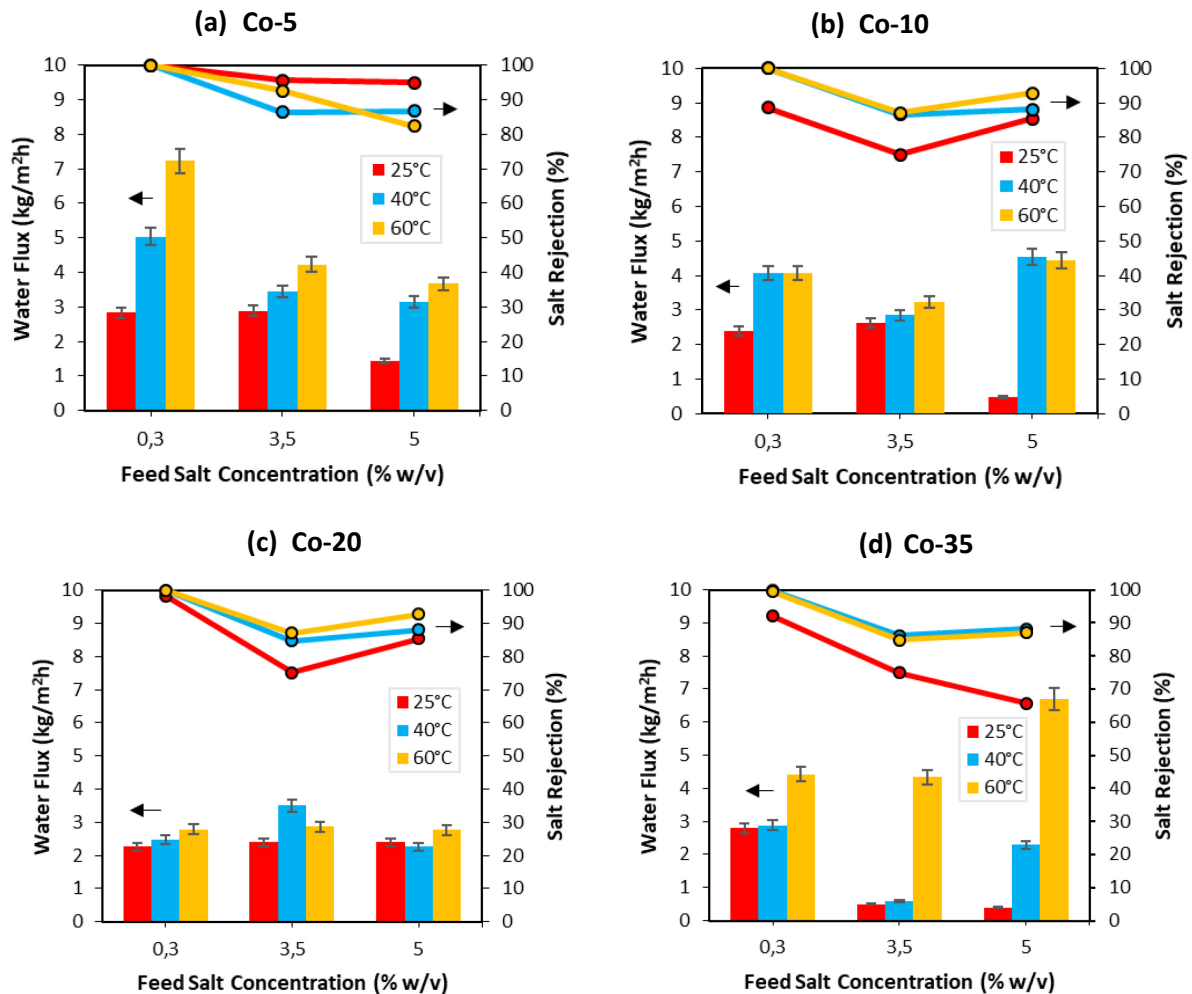


Fig. 5. Relation between water flux and salt rejection on various membranes in various feed salt concentration and temperatures

Conclusions

The conclusion of this work is that the higher concentration of cobalt in the membrane, will increase silanol and siloxane groups and increase the pore size of membranes. In addition, the value of water flux will tend to decrease but salt rejection will increase. On the other hand, the increasing concentration of NaCl in the feed will also reduce the value of water flux and increase salt rejection. While the influence of operating temperature is the higher operating temperature, the water flux will increase.

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Performance of Cobalt-Silica Membranes Through Pervaporation Process with Different Feed Solution Concentrations

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Abstract. This work shows the performance of cobalt-silica membranes through water desalination via pervaporation process. The aim of this work is to find out the performance of the cobalt oxide as a templating agent in the silica cobalt membranes for water desalination via pervaporation process. It also aims to investigate the water flux and salt rejection of silica cobalt membranes using artificial saline water. The concentration of cobalt oxide as a template for fabricating cobalt-silica membranes were 5 – 35 wt%. The feed solution applied during pervaporation process were 5 – 5 wt% NaCl with operating temperatures of 25, 40 and 60 °C. The silica xerogels were characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM) and Brunauer-Emmett-Teller (BET). From the FTIR result, it is known that the higher the concentration of cobalt in the membrane, the more silanol and siloxane groups. Based on BET analysis, Si-Co 35 wt% membrane has largest pore volume (0.129387 cm³g⁻¹). In addition, the highest value of water flux (7.2660 kg.m⁻².h⁻¹) and salt rejection (100%) is achieved by Si-Co 5% membrane in 0.3% NaCl feed at 60 °C. The value of water flux decreases and the value of salt rejection increases with increasing feed concentration.

Introduction

Increasing sea level as a result of climate change causes less fresh water to be used as potable water. Therefore, the source of drinking water from seawater, a process called desalination, is a logical way forward to address the water needs of current and future needs. Seawater desalination can be done by membrane separation processes such as reverse osmosis (RO), membrane distillation (MD) or pervaporation (PV).

Membrane technology has long been a separation process in the industrial world because of its fast and efficient process. In general, there are three types of membrane processes that are usually applied, it is called reverse osmosis (RO), membrane distillation (MD) and pervaporation (PV)[1]. Membrane is basically a thin layer between two fluid phases, its feed phase and the permeate phase which is a barrier to a particular species, which can separate substances of different sizes and limit the transport of various species based on physical and chemical properties. Membranes are semipermeable, mean that membrane can hold certain species larger than the membrane pore size and pass other species of smaller size. The selective nature of this membrane can be used in the separation process. Membrane support is a membrane from ceramic material made through high temperature combustion[2]. The stability of membrane strength depends on the chemical, mechanical and thermal conditions of the membrane[3]. Membranes in their application for water treatment do not require the addition of chemicals (coagulants, flocculants) so that they are friendly to the environment[4].

Inorganic membranes have recently caught the attention of the research and industry communities because of their long life span and better resistance to environmental factors and extrinsic industries. Now, there are two types of inorganic silica membranes which have been being

widely observed for water desalination, they are zeolite and amorphous silica membranes. Zeolite membranes have proven effective but long-term stability and improvement still present problems to date. Silica membranes have excellent molecular filtration properties and simple fabrication processes through sol-gel processing, although they need functionalization to overcome hydro-instability. The structure of the silica membrane has a pore size in the range of 3-5 Å while the kinetic diameter of water molecules is 2.6 Å, making it ideal for inhibiting the passage of hydrated salt ions (Na^+ : $d_k = 7.2 \text{ \AA}$ and Cl^- : $d_k = 6.6 \text{ \AA}$). Therefore, the silica membrane separates water from salt with a molecular filtration mechanism.

Methodology

Thin Film Fabrication and Pervaporation. Silica sols were synthesized from a two-step sol gel process employing acid and base as catalysts (followed our previous work [5]). Tetraethyl orthosilicate (TEOS) was added drop-wise into ethanol (EtOH) solution and stirred for 5 minutes in ice bath condition at 0 °C to avoid partial hydrolysis followed by the addition of diluted nitric acid (0.0008 M HNO_3). The sol mixture was refluxed for 1 hours at 50 °C with stirring to achieve a complete hydrolysis of the alkoxy groups. Ammonia solution (NH_3) diluted in ethanol was added drop wisely into sol mixture to commence the condensation reaction and reflux was continued for another 2 hours to obtain the resultant sol. Silica sols solution was then dried in a temperature controlled (oven) at 60 °C for 24 hours to obtain the dried gel for xerogel characterization. The final molar ratios of the sol (TEOS:EtOH: HNO_3 : H_2O : NH_3) were calculated to be 1:38:0.00078:5:0.003. Then, silica sol mixed with cobalt oxide 0.2873 g for Si-Co 5%, 0.5746 g for Si-Co 10%, 1.1492 g for Si-Co 20% and 2.0111 g for Si-Co 35%. Furthermore, Si-Co sol solution (thin membrane films) were coated on macroporous alumina substrates ($\alpha\text{-Al}_2\text{O}_3$ tubular support, pore size $\approx 100 \text{ nm}$) via a dip-coating process with a dwell time of 2 minutes and a dipping and withdrawal rate of 10 and 5 $\text{cm}\cdot\text{min}^{-1}$, respectively. After the deposition of each layer of thin film onto membrane support, it was then dried and calcined in air at 600 °C for 1 hours. This cycle of dip-coating and calcination process was repeated up to four layers. Next step is to prepare a feed solution of 0.3; 3.5; 5% of artificial saline water. Each feed solution was placed into the feed tank. Then connect the cold trap and assemble in the classical pervaporation set-up as shown in figure 1. The dead-end mode of tubular Si-Co membrane was set (one of end of tube was blocked and the other was connected to cold trap and vacuum pump). Membrane was immersed into feed solution and then the pervaporation was started in every 20 minutes. The separated solution will evaporate and flow through pore size of membrane and collected in the cold trap. The evaporated molecules was then condensed in the cold trap by immersing cold trap into liquid nitrogen. The following step is to calculate the water flux and measure the salt rejection using a conductivity meter. Then calculations of the water flux and salt rejection were repeated three times in every measurement. The water flux, F ($\text{kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$), was determined based on the equation

$$F = m/(A \cdot \Delta t) \quad (1)$$

where m is the mass of permeate (kg) retained in the cold trap, A is the surface-active area (m^2) and Δt is the time measurement (h). The salt rejection, R (%), was calculated as

$$R = (C_f - C_p)/C_f \times 100\% \quad (2)$$

where C_f and C_p are the feed and permeate concentrations of salt (wt %)

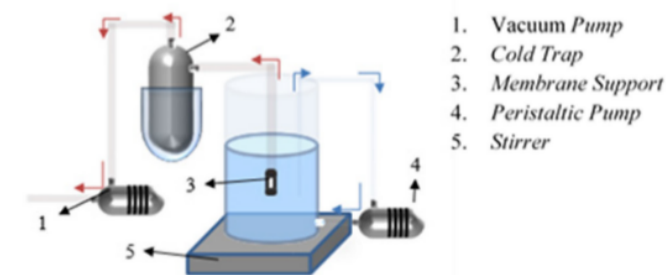


Fig. 1. Desalination rig via classical pervaporation set up

Result and Discussion

Xerogel Characterization. Silica membranes are fabricated from silica synthesis with the sol-gel method that has been developed mainly for gas separation with pore sizes in the range of 3-5 Å[6]. The difficulty of using silica membranes for desalination is related to the instability of the silica matrix when exposed to water[7]. In order to deal with this problem, researchers have implanted and carbonated with covalent templates or surfactants[8] in the silica matrix. Recently, a number of research groups reported the development of metal or metal oxide doping on silica membranes with exceptional gas selectivity, thus showing good pore size reduction. One of the metal oxides is cobalt [9, 10]. In preparing cobalt-silica sol is by applying the sol-gel method, the sol was obtained in this study where soles with a pH of 6 would produce mesoporous membrane pores[5]. It is in order to fabricate the pore size of silica matrices were greater than the pore size of a water molecules. Also, it is smaller than salt molecules. This supposed to water molecules are easily sieved through silica matrices. Literally, silica sol is an aqueous clear colorless liquid, where after adding it to cobalt the color will turn clear purple. In addition to providing strength to the membrane pores, the addition of cobalt in silica sol will affect silanol and siloxane groups. Silanol and siloxane groups are known to affect the membrane pore size, so it is important to know whether or not there are two groups[11]. Using the Fourier Transform Infrared (FTIR) analysis method, it can be seen the area of silanol and siloxane obtained in cobalt-silica sol. From the results of the FTIR analysis the graph is shown in Figure 2(a).

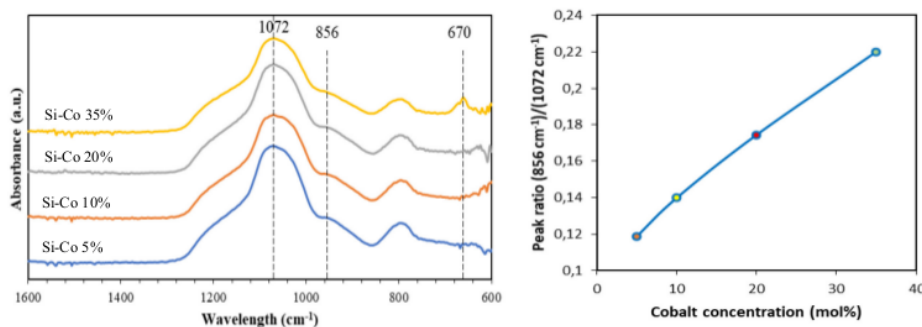


Fig. 2. (a) FTIR spectra of cobalt-silica xerogels, (b) deconvulation data between silanol and siloxane of cobalt-silica xerogels

Figure 2(a) shows the peaks of siloxane groups found in the wavelength 1072 cm^{-1} and 798 cm^{-1} . and the peaks found in the wavelength 856 cm^{-1} is silanol groups. Then, also found Co_3O_4 in the wavelength 607 cm^{-1} . These peaks are slightly similar from previous studies [11]. In figure 2(b) tells about the deconvulation data. Deconvulation data were observed from Fityk software from wavelength 600 to 1600 cm^{-1} . The error of this Fityk software analysis was investigated for about

5%. Gaussian curve was applied to fit the curves. Also, In order to deconvolute peaks, HWHM (the half width half max) has been done for every single peaks. And then, the area of silanol group (856 cm^{-1}) was compared to siloxane groups (1072 cm^{-1}) as well as the concentration of cobalt oxide in the calcined samples (silica xerogels). From the figure 2(b), it can be seen that the deconvoluted area increases with the increasing of the concentration of cobalt in the samples. Moreover, when the concentration of cobalt oxide templated into silica sols increase, so, the silanol groups in the silica matrix also increase while the siloxane groups decrease. This is correlated to the results of Elma's study [11] which also states that the concentration of silanol groups in the silica matrix increases with increasing concentration of cobalt oxide in the silica matrices.

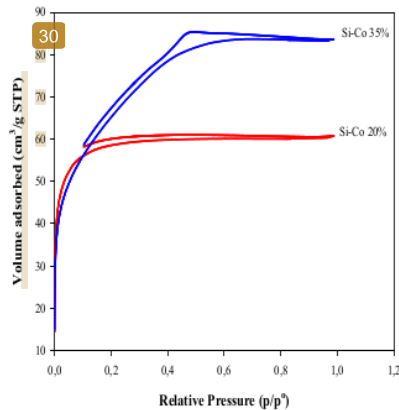


Fig. 3. Plots of N_2 isotherm data of the calcined Co-Si xerogels

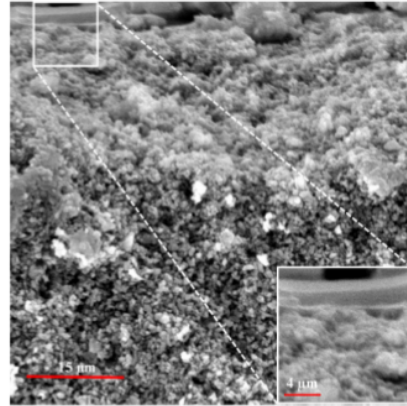


Fig. 4. SEM image cross-section Si-Co 5% membrane

Figure 3 is a graph of isothermal N_2 adsorption on Si-Co xerogels with the concentration of cobalt oxide 20 wt% and Si-Co xerogels with the concentration of cobalt oxide 35 wt%. Based on the results of BET analysis, Si-Co xerogels with the concentration of cobalt oxide 20 wt% has a lower surface area ($188.9547\text{ m}^2\text{g}^{-1}$) than Si-Co xerogels with the concentration of cobalt oxide 35 wt%. Also, it is with a total volume of $0.094015\text{ cm}^3\text{g}^{-1}$. While Si-Co xerogels with the concentration of cobalt oxide 35 wt%. has a high surface area of $225.3542\text{ m}^2\text{g}^{-1}$ with a total volume of $0.129387\text{ cm}^3\text{g}^{-1}$. In addition, the pore size of the Si-Co xerogels with the concentration of cobalt oxide 20 wt% is smaller than the pore size of the Si-Co xerogels with the concentration of cobalt oxide 35 wt%. It is due to the cobalt oxide create the pore size bigger. The higher the content of cobalt oxide in xerogels, the bigger the size of the pores. In a previous study [11] a silica xerogels produces also found in mesoporous size.

Figure 4 is obtained through a SEM (Scanning Electron Microscopic) which shows a cross section of Si-Co membrane 35% which is made through the process of dipcoating and rapid thermal process (RTP). In the first dipcoating process, the thin film then fill the pores of the membrane support which has a big pores ($\sim 100\text{ }\mu\text{m}$), so that the sols will completely meet the membrane pores of support. During the dipcoating process, after the membrane support pores have been filled, then the thin film layer begins to form. And this process will continue up to fourth layer. From figure 4 found that the membrane thickness of Si-Co membrane with the cobalt concentration 35% was $1.9\text{ }\mu\text{m}$. After dipcoating, the membranes were calcined using rapid thermal processing (RTP) technique for 1 h at $600\text{ }^\circ\text{C}$. The calcination process of membranes done after dipcoating process was held in every layer. It is found that silica membranes fabricated through this rapid thermal process (RTP) are thicker than silica membranes which are processed through a conventional calcination processing (CTP) technique. It is because the calcination process using RTP technique is much shorter than (1 h) the membranes fabrication using CTP technique (4 h). It may also cause the solvent from thin film dipcoated onto membrane support is trapped inside the silica matrix during

calcination process, so that the evaporation process during calcination process may be incomplete. The calcination process carried out directly from the room temperature at high temperatures cause the ethanol as a solvent is not completely evaporated. Then, there is still H₂O molecules remained in the silica matrices. This is phenomena is addressed to describe the membranes thickness after fabrication. When compare to the thickness of the membrane fabricated by the conventional calcination processing (CTP) techniques investigated from Elma [11] was 0.4 μm. Interestingly, even though the silica cobalt membranes fabricated from RTP technique is thicker than silica cobalt membranes fabricated from CTP technique, the performance of these membranes is excellent. It then can be seen from the high value of salt rejection in Figure 5.

Water Flux and Salt Rejection. Figure 5 shows the relationship between water flux and salt rejection on various types of silica cobalt membranes with various types of feed solution. As we know that water flux is the ability of a membrane to be passed by water molecules per unit area per unit time. Whereas salt rejection is the ratio between the salt concentration that does not through the membrane pores and the salt concentration initially at the feed. The value water flux is obtained from the mass of water obtained in the cold trap divided by the surface area of the membrane in contact with the feed and divided by the length of time pervaporation. In 5% Si-Co membrane, the highest water flux is obtained at a 0.3% NaCl feed temperature of 60 °C which is 7.2660 kg.m⁻².h⁻¹ with 100% salt rejection. In 10% Si-Co membrane, the highest water flux is obtained at 10% NaCl feed temperature of 40 °C, which is 4.5383 kg.m⁻².h⁻¹ with salt rejection 88.14%. In the 20% Si-Co membrane, the highest water flux was obtained at a 3.5 wt% NaCl feed temperature of 40 °C which was 3.4951 kg.m⁻².h⁻¹ with salt rejection 84.62 %. In 35 wt% Si-Co membrane, the highest water flux was obtained at a 5% NaCl feed temperature of 60 °C which is 6.6842 kg.m⁻².h⁻¹. From figure 5 also can be seen that water flux decreases with the increasing of feed concentration. In addition, silica cobalt membranes with a high concentration of cobalt also give low water flux compared to water flux in membranes with lower concentrations of cobalt as a templating agent. The highest water flux value of all silica cobalt membranes found in 0.3 wt% NaCl feed. This is due to small amount of salt particles that clog the pores of the membrane itself. So that the value of the water flux obtained become high. The decrease of water flux is caused by the increasing number of salt particles in the feed solution. It is caused by the pores of the membrane become more clogged. While the decrease in water flux to increase the concentration of cobalt in the membrane due to the increasing number of silanol groups along with the increasing concentration of cobalt in the membrane, so more salt particles of water molecules are trapped inside silica matrices. The silanol group is the bond between silica and the -OH group, which cause the silanol pore size to be smaller than the siloxane pore size. -OH which comes into contact with water molecules (H₂O) become more active and give effect to the membrane performance become collapse. The ruins widen the pore at the top but get narrower at the bottom due to the clogged debris. The pore forms becomes an inverted cone shape. It also causes the value of water flux obtained in this study to be lower than that of water flux obtained by Elma [11] due to more concentration of silanol groups in the thin film. The value of salt rejection on each membrane tends to decrease with increasing salt concentration in the feed. Many salt particles clog the pore, with the high thrust the particles are increasingly trying to penetrate the pore, causing damage to the pore and salt particles to escape.

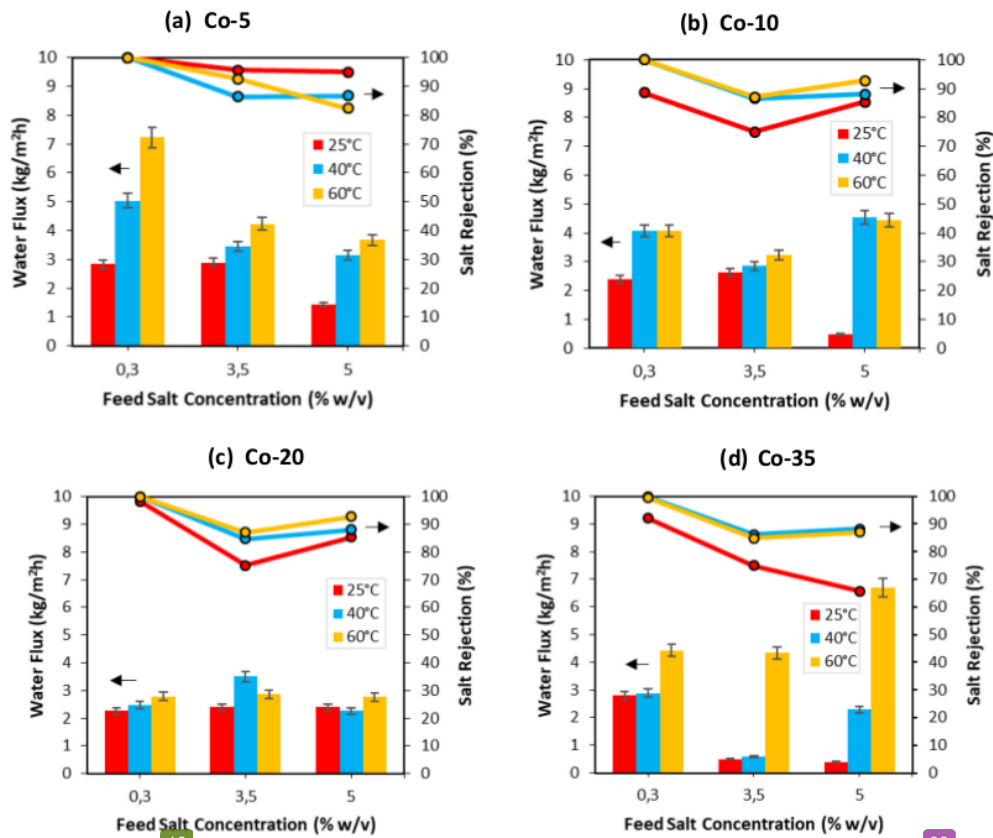


Fig. 5. Relation between water flux and salt rejection on various membranes in various feed salt concentration and temperatures

Conclusions

The conclusion of this work is that the higher concentration of cobalt in the membrane, will increase silanol and siloxane groups and increase the pore size of membranes. In addition, the value of water flux will tend to decrease but salt rejection will increase. On the other hand, the increasing concentration of NaCl in the feed will also reduce the value of water flux and increase salt rejection. While the influence of operating temperature is the higher operating temperature, the water flux will increase.

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