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TECHNOLOGY

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ISSN: 0958-2118

Membrane Technology

Country	Netherlands - SIR Ranking of Netherlands
Subject Area and Category	Agricultural and Biological Sciences Food Science Chemistry Chemistry (miscellaneous) Energy Energy Engineering and Power Technology Engineering Electrical and Electronic Engineering Industrial and Manufacturing Engineering Environmental Science Environmental Science (miscellaneous) Materials Science Materials Science (miscellaneous)
Publisher	Elsevier BV
Publication type	Journals
ISSN	09582118
Coverage	1991-ongoing
Scope	Membrane Technology brings you everything you need to keep up-to-date with what is happening in the international membrane industry, highlighting emerging markets, summarising important industry news and identifying new business opportunities for your company. Each issue includes: • Technology news - all the latest developments including processes projects, contracts, systems, new products, testing facilities, etc. • Business news - key information on your competitors movements in the market • Feature articles - experts share the secrets of their own success • Patents - the latest new patents from around the world • Research trends - highlights of recently published research work
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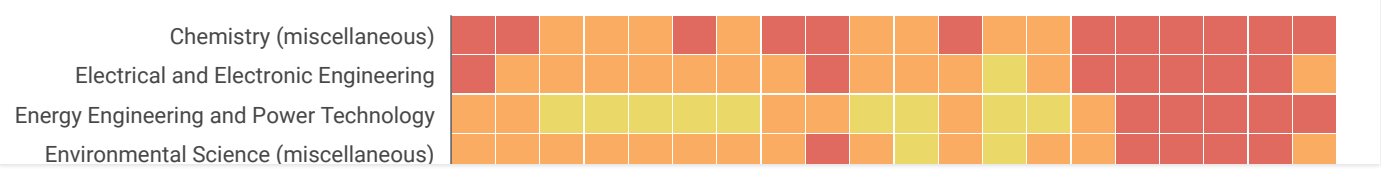

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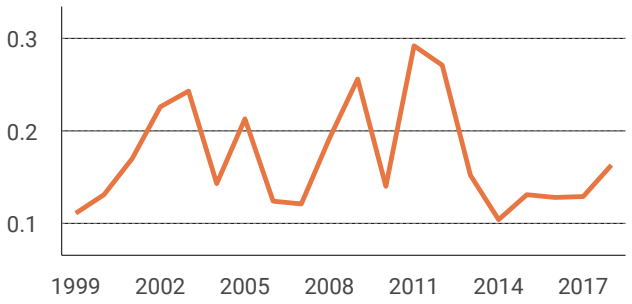

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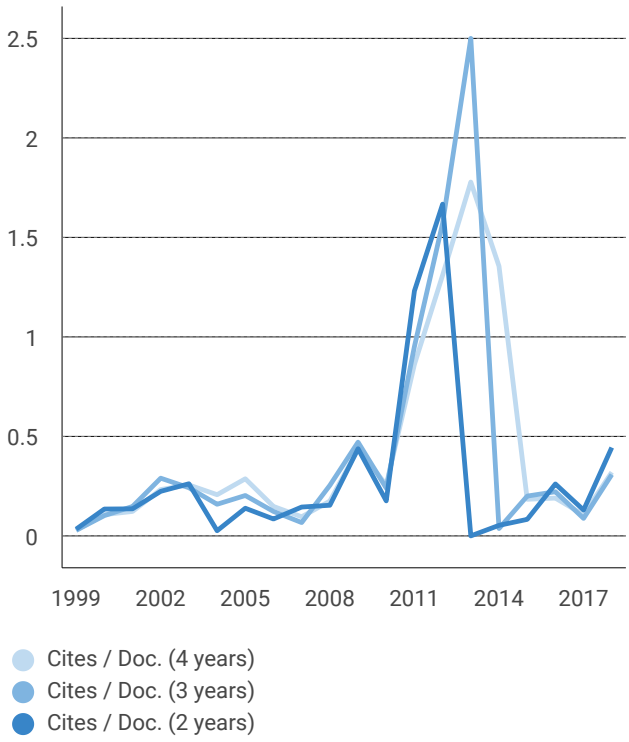

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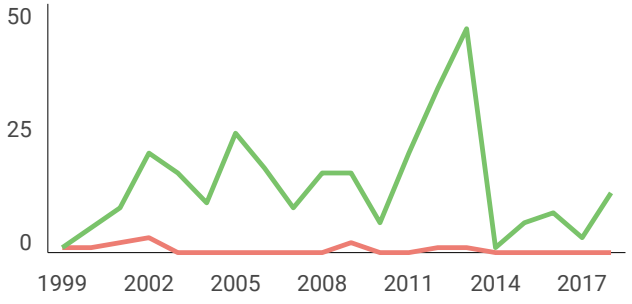

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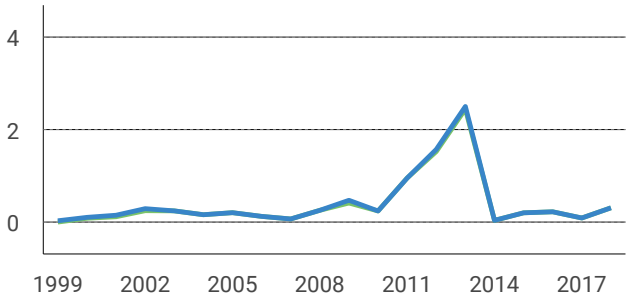

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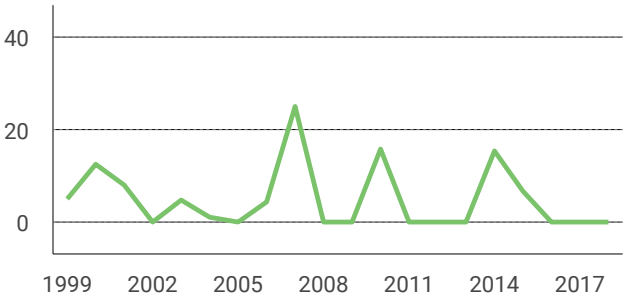

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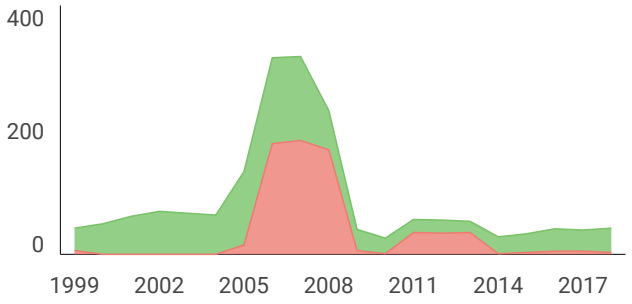

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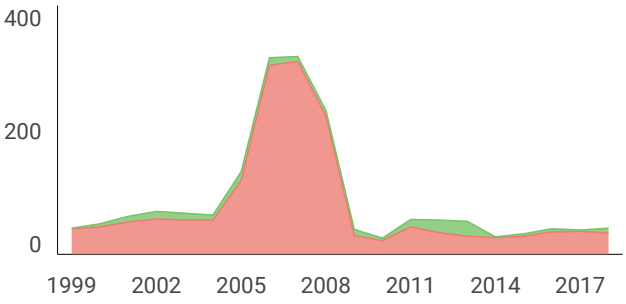

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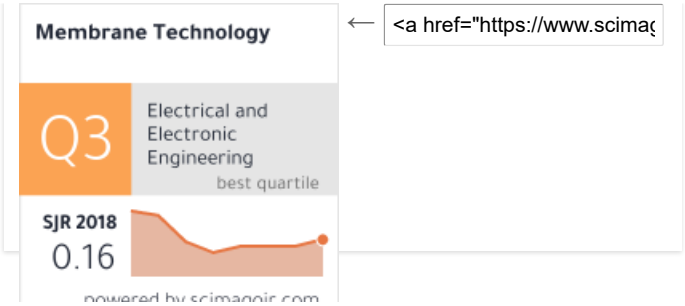

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ISSN: 0

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Membrane Technology

Volume 2019, Issue 12, December 2019, Pages 5-9

Feature

Interlayer-free silica–pectin membrane for sea-water desalination

Erdina L.A. Rampun, Muthia Elma, Aulia Rahma, Amalia E. Pratiwi

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https://doi.org/10.1016/S0958-2118(19)30222-8

Water scarcity has become a serious problem worldwide. Research is now looking at the fabrication and use of interlayer-free silica–pectin membranes for sea-water desalination as one potential solution to this problem. This research work investigates the performance of interlayer-free silica–pectin membranes for artificial sea-water desalination. A dual acid–base catalyst process is applied during the sol–gel process used to prepare the membrane's thin layer structure. Membranes were prepared using tetraethyl orthosilicate as the silica precursor and various concentrations of pectin (0 wt%, 0.1 wt%, 0.5 wt% and 2.5 wt%) were used as the "templating" agent. The performance of membranes was evaluated using pervaporation (PV) processes at room temperature (~25°C). The results show that water flux for 0.1 wt% pectin was the highest (4.50 kg m^{-2} h^{-1} and 5.73 kg m^{-2} h^{-1}, at calcination temperatures of 300°C and 400°C, respectively). The fluxes at 0.5 wt% and 2.5 wt% pectin concentration were much lower, compared with 0.1 wt% (3.12–0.51 kg m^{-2} h^{-1} for both, at calcination temperatures of 300°C and 400°C). In addition, the salt rejection for all membranes remained excellent, at ~99.9%. This clearly demonstrates that carbon chains in silica matrices may produce smaller and stronger pores.

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Interlayer-free silica–pectin membrane for sea-water desalination

Erdina L.A. Rampun, Muthia Elma, Aulia Rahma and Amalia E. Pratiwi, Chemical Engineering Department, Engineering Faculty – and Materials and Membranes Research Group (M^2ReG) – Lambung Mangkurat University, Banjarbaru, South Kalimantan, Indonesia

Water scarcity has become a serious problem worldwide. Research is now looking at the fabrication and use of interlayer-free silica–pectin membranes for sea-water desalination as one potential solution to this problem. This research work investigates the performance of interlayer-free silica–pectin membranes for artificial sea-water desalination. A dual acid–base catalyst process is applied during the sol–gel process used to prepare the membrane's thin layer structure. Membranes were prepared using tetraethyl orthosilicate as the silica precursor and various concentrations of pectin (0 wt%, 0.1 wt%, 0.5 wt% and 2.5 wt%) were used as the "templating" agent. The performance of membranes was evaluated using pervaporation (PV) processes at room temperature (~25°C). The results show that water flux for 0.1 wt% pectin was the highest (4.50 kg m^{-2} h^{-1} and 5.73 kg m^{-2} h^{-1}, at calcination temperatures of 300°C and 400°C, respectively). The fluxes at 0.5 wt% and 2.5 wt% pectin concentration were much lower, compared with 0.1 wt% (3.12–0.51 kg m^{-2} h^{-1} for both, at calcination temperatures of 300°C and 400°C). In addition, the salt rejection for all membranes remained excellent, at ~99.9%. This clearly demonstrates that carbon chains in silica matrices may produce smaller and stronger pores.

The water crisis afflicting many counties is one of the most serious problems facing communities worldwide. As populations throughout the world continue to grow this problem becomes more pressing.

To help find a solution to this problem, new approaches need to be developed to satisfy the increasing demand for clean water. Vast amounts of sea water are seen as a potential water resource.

Recently, membrane desalination using pervaporation (PV) has become a most favourable technique to produce potable water. Many pervaporative desalination studies have been done and appear in the literature.[1–5] PV has also been used to treat various salt concentrations without the need to set the pressure difference.[6]

Reverse osmosis (RO) requires the use of a high-pressure pump to treat a high salt concentration.[7]

In PV, the "driving force" behind the membrane is the chemical potential gradient between the feed and permeate. It is obtained from the difference in the partial pressures of the components on the two sides of the membrane.[8] PV has several advantages, such as a high rejection, a low operating cost and only requires low pressure.[9]

Silica

There is growing interest in using silica-based membranes for pervaporation. This is because during operation they are thermally stable.[10]

Unfortunately, obstacles to using silica membranes relate to their low hydro-stability[11] when in contact with water molecules. The concentration of silanol (Si–OH) groups in these membranes[12] also affects their properties.

Silanol is hydrophilic and when exposed to water will result in an enlargement in the pore size. It may affect membrane performance.[13]

Surface and pore silica can be easily controlled by using the sol–gel method.[14] It is also possible to modify silica and increase stability with a carbon template,[13, 15 & 16] through hybrid organic–inorganic structures[17–19] and using a metal oxide.[20–23]

Silica–carbon membranes have been fabricated successfully using the triblock copolymer Pluronic® P123 as the template. Results show that water flux of 2.5 kg m^{-2} h^{-1} was achieved for sea-water desalination.[16] However, the use of carbon from the P123 material brings with it relatively high costs.

On other hand, a sustainable carbon precursor such as pectin has good properties that enable it to form gel.[24] In addition, it is inexpensive and an environment-friendly material. It can be obtained from orange peel,[25] apple peel[26] and banana peel.[27]

The downside is that pectin films have poor thermal stability and mechanical properties.[28 & 29] Therefore, it was selected as a template along with silica.

"Interlayer-free"

Lately, new membrane structures without interlayers have been developed. Referred to as "Interlayer-free", they are created by dip-coating sols directly onto an alumina substrate.[16 & 20]

Other work has covered generally fabricating membranes that comprise a support, interlayers and thin film in multilayers. By disregarding the interlayers the thickness of the membrane can be reduced and its performance may be improved.

For instance, the water flux and salt rejection of pure silica membranes fabricated without and with an interlayer are (8.5 kg m^{-2} h^{-1}; > 99.5%) and (2.3 kg m^{-2} h^{-1}; 91%), respectively.[13 & 16]

In addition, when comparing work that involves conventional thermal processing (CTP) and rapid thermal processing (RTP), the fabrication of membranes using CTP takes longer (in excess of one week). This is because of the slow calcination process and the need to allow 1°C min^{-1} for dwelling time.

Producing membranes using RTP takes a short period – less than one day of production time.[30] For this reason, in this work, we employ the RTP method to produce membranes.

In addition, we also use pectin as a sustainable carbon material instead of synthetic carbon,[31] and focus on investigating the performance of interlayer-free silica–pectin membranes for artificial sea-water desalination.

Experimental work

Chemicals and materials

The experimental work made use of the following materials:

- tetraethyl orthosilicate (TEOS), 99.0% (Sigma-Aldrich);
- pectin from apple (Sigma-Aldrich);
- ethanol (EtOH), 99%;
- aquadest;
- dilute nitric acid (HNO_3), 0.0008 M (Merck);
- ammonia (NH_3), 0.0003 M (Merck);
- glycerol, 85% (Merck);
- a tubular membrane support, Al_2O_3 (Ceramic Oxide Fabricators, Australia), with a length of 50 mm and outside diameter of 8 mm;
- 3.5 wt% NaCl as the feed solution; and liquid nitrogen.

Fabrication and characterisation

Sol–gel was prepared using dual acid–base catalysts by adding TEOS, drop-wise to the ethanol (EtOH, 99%), whilst stirring the solution for 5 min at a temperature of 0°C. HNO_3 was then added to the solution and refluxed for 1 h at 50°C.

This process was followed by adding NH_3, dissolved in ethanol, and the solution was stirred for another 2 h under the same conditions. The pH of the final sols was checked (pH 6).

Pectin (0.1 wt%) was dissolved into 5 ml of glycerol whilst stirring at 360 rpm for 45 minute at 40°C. This mixture was added to the pure silica sol and again stirred at 360 rpm for 45 minute at 0°C. The sol was dried in oven at 60°C to produce xerogels. After that, the xerogels were calcined in air at 300°C and 400°C.

The initial molar ratios of silica–pectin – TEOS : EtOH : HNO_3 : H_2O : NH_3 : pectin – were calculated to be 1:38 : 0.0008 : 5 : 0.00 03 : 0.000026.

Membrane preparation and pervaporation testing

Membranes were prepared via dip-coating.

The tubular support (α-Al_2O_3, with an average pore size 100 nm) was dip-coated four times using the prepared silica–pectin sol. It was calcined in air at 300°C and 400°C for 1 h using RTP. The morphology was observed using scanning electron microscopy (Zeiss), looking at a cross-sectional area and the surface of the membrane.

Membrane testing was accomplished using the pervaporation set-up that is shown in **Figure 1**.

Sea water (3.5 wt%) was prepared and used as the feed solution at room temperature (25 ± 2°C). The feed solution was vaporised and passed through the membrane. The vacuum pump (operating at ~1 bar pressure) was used to maximise the driving force.

Finally, the vaporous permeate was condensed, using liquid nitrogen, to form a liquid. The feed solution was stirred to avoid concentration polarisation. The water flux F (kg m^{-2} h^{-1}) was calculated using **Equation 1**.

$$F = [m/(A \times \Delta t)]\% \quad (1)$$

where m is mass of permeate (kg) retained in the cold trap; A is the active surface area of membrane (m^2); and Δt is the collection time (h).

The salt rejection R (%) was calculated using **Equation 2**.

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

where C_f and C_p are the feed and permeate salt concentration, which were determined using a conductivity meter SF300C-G (Ohaus).

Results and discussion

Silica–pectin membrane morphology

Membrane morphology was determined using scanning electron microscopy (**Figure 2a** and **Figure 2b**) in order to gain further understanding of the effect of morphology on membrane performance. The thickness of the interlayer-free silica–pectin membrane was around 916.3 nm.

This membrane is quite thick because of the rapid calcination process that was used. Silica–carbon sols penetrated the alumina support during the dip-coating and calcination processes.[16] This was driven by the high capillary forces present during the repeated application of the coating (four-layers) and non-uniform thickness of the thin film was observed throughout the membrane.

However, no cracks were found on the surface of silica–pectin thin film (Figure 2b).

The presence of pectin could be increasing the sol viscosity when the infiltration process is taking place. Therefore, the interlayer-free structure produces stronger and thicker pores. This is also confirmed by the excellent salt rejection (99%). In addition, as shown in our previous work, RTP results in a thicker film, compared with the CTP (470 nm).[32] The

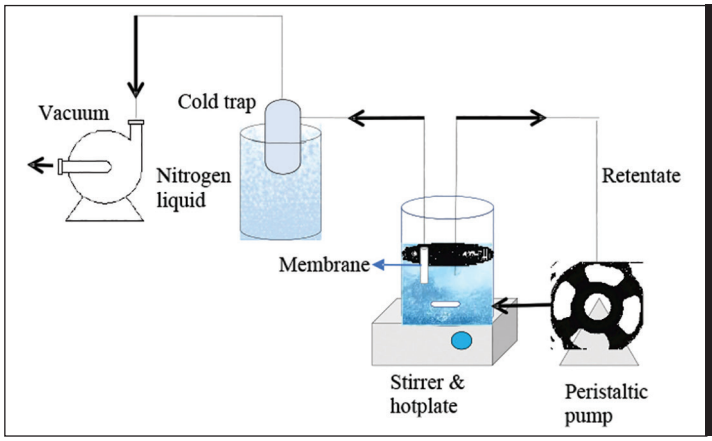

Figure 1. Pervaporation set-up for sea-water desalination.

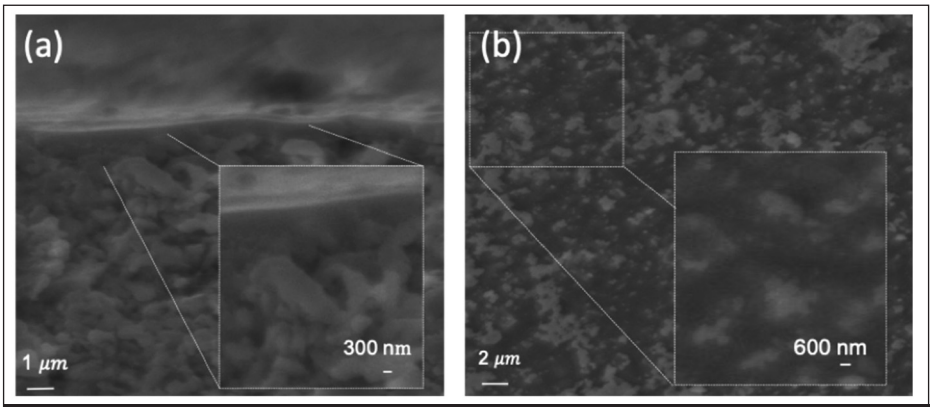

Figure 2. Scanning electron microscopy of an interlayer-free silica–pectin membrane during calcination in air: cross-sectional area (a) and surface area (b).

absence of fast calcination and ramping rate in RTP results in imperfect solvent evaporation.

Membrane performance

Figure 3 shows the performance of the inter-layer-free silica–pectin membrane using artificial sea water (3.5 wt%) as the feed solution.

As can be seen, this work demonstrates that it is possible to achieve a high water flux (5.73 kg m^{-2} h^{-1}) and excellent salt rejection (> 99.9%). It clearly shows the viability of the silica–carbon membrane which uses pectin as the carbon source.

Furthermore, the promising carbon-derived material enhances silica hydro-stability for sea-water desalination. Indeed, even with a low concentration of pectin (0.1 wt%) excellent performance is still achievable. This finding has the potential to reduce the cost of membrane fabrication by using a small quantity of materials.

This work has addressed the effect of pectin concentration on the silica matrix (**Figure 4**). Firstly, water flux increases sharply when pectin is used as template in the silica material (0.1 wt%).

However, when the pectin concentration is increased further, performance and water flux appear to decline. This may be because larger amounts of pectin, as the carbon concentration, can lead to membrane densification.

This means that in this case there is a limit to the increase in pectin concentration. The carbon number for a higher pectin concentration will result in the formation of non-ionic micelles, which blend with the silica network.[16]

Contrary to this, our previous study (done by Elma *et al.*[16]) stated that increasing carbon content would result in the formation of a mesoporous structure (2–50 nm). Based on that, we assume that the number of carbon chains in the pectin is much lower than that in Pluorionic P123. However, the salt rejection for all of the membranes remains constant at > 99%.

In addition, Figure 3 shows the effect of temperature when RTP is used. Increasing the calcination temperature does not have much impact on water flux for a higher pectin concentration (0.5 wt% and 2.5 wt%). But interestingly, it delivered a high water flux on a lower pectin concentration (0.1 wt%) and great salt rejection.

For this reason, it is suggested that volumetric strain rises when the temperature increases. This opinion totally agrees with a previous study,[33] though other studies have also found that RTP produces a more non-porous membrane than CTP.[34] Despite the latter the use of RTP is preferred in order to reduce fabrication time.

The flux achieved in this research competes relatively well with that reported in other work, as shown by the studies listed and summarised in **Table 1.** So far, the three highest water flux

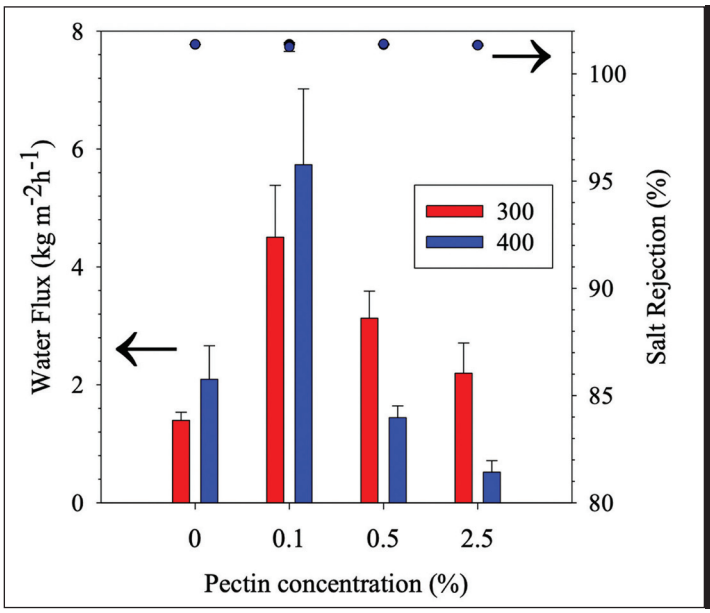

Figure 3. Effect of pectin concentration and calcination temperature on membrane performance for sea-water desalination.

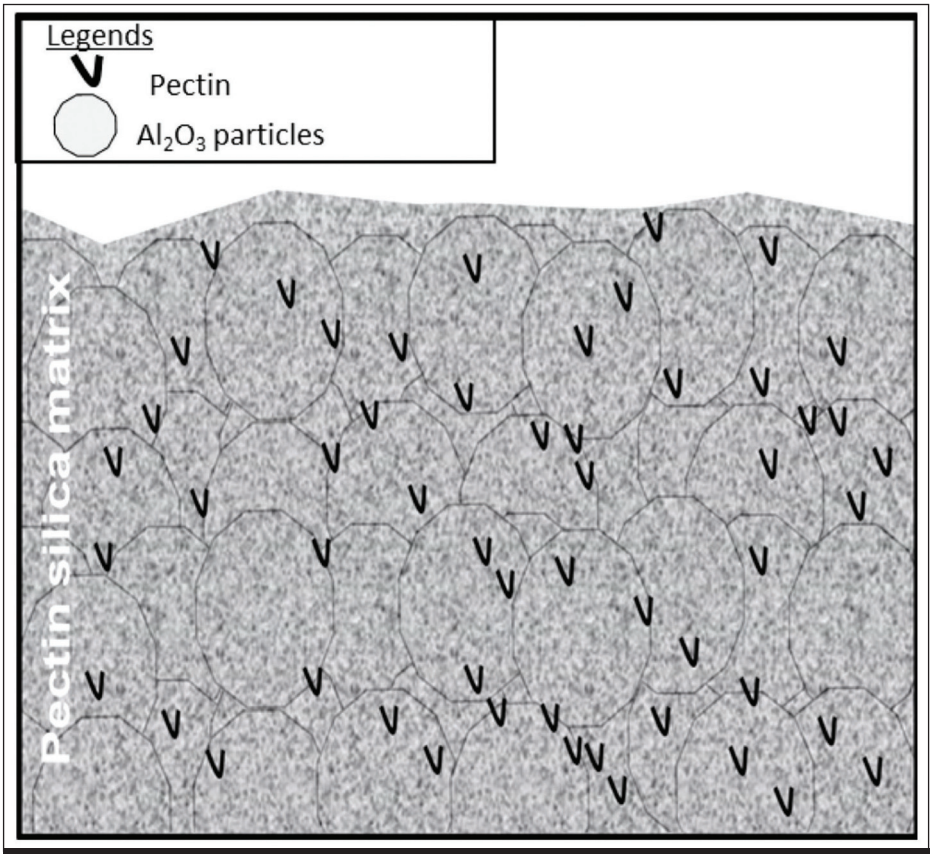

Figure 4. Illustration of pectin (0.1 wt.%) "attached" to silica on a membrane support.

figures – 20.00 kg m^{-2} h^{-1}, 19.3 kg m^{-2} h^{-1} and 17.8 kg m^{-2} h^{-1} – were achieved by Song *et.al.*, Yang *et. al.* and Wang *et.al.*

As the table reveals, different types of membranes, such as carbon–alumina mixed matrix, were used. In addition, a different precursor, ethyl silicate (ES40), was also used and all of them employed RTP. This led to greater water loss and solvent evaporation inside the xerogel because of the higher calcination temperature and formation of siloxane bridges.[33]

Besides that, our work produced high values, conducted under a feed temperature of 25°C (Table 1).[13, 16 & 35–37] Here, the acid–base catalysts are playing an important role in controlling the pore size of the membrane. Acid catalysts form small pores, whereas base catalysts promote a mesoporous structure.[38]

Small pores are certainly able to achieve a high rejection, but result in a lower flux and, vice versa, the mesoporous structure produces lower salt rejection and higher flux values.

Taking this into consideration, it is a good idea to work with a combination of a small and mesoporous construct by employing acid and base catalysts, to create a mix of a small and

Membrane type	Calcination technique	Feed temperature (°C)	Feed concentration (%)	Water flux (kg m^{-2} h^{-1})	Rejection (%)	Reference
Silica–pectin	RTP	25	3.5	5.73	> 99	(this work)
Carbon template silica	CTP	22	3.5	2.2	> 99	[16]
Carbon alumina mixed matrix	RTP	25	3.5	20	> 99	[40]
Carbon silica membrane	RTP	60	3.5	19.3	99	[17]
Ethyl silicate (ES40) membrane	RTP	60	3.5	17.8	> 99	[33]
Triblock copolymer templated silica membrane	CTP	25	3.5	3.7	98.5	[35]
Molecular sieve silica (CTMSS)	CTP	25	3.5	1.9	98	[36]
Mesoporous silica	CTP	22	3.5	6.8	> 98	[32]
Silica–TEVS membrane	N/A	22	3.5	< 2	> 99	[37]

Table 1. Comparison of silica–carbon membrane performance using rapid thermal processing (RTP) and conventional thermal processing (CTP), for desalting sea water.

mesoporous structure, referred to as a "bottle neck".[32]

In principle, water passes through membrane, but the combined pores avoid Na^+ and Cl^- release with the permeate, which have larger molecules. But this still offers a high water flux because of the mesoporous component itself.

In addition, our recent study using a single catalyst (for example, citric acid) to form a mesoporous structure, has been successful.[39]

Conclusion

In this study we have demonstrated interlayer-free silica–pectin fabrication and successfully delivered high water fluxes (4.50 kg m^{-2} h^{-1} and 5.73 kg m^{-2} h^{-1}, for materials calcined at 300°C and 400°C, respectively).

This is a significant achievement because pectin plays an important role in creating stronger pores, as evidenced by the salt rejection which remained at 99%.

In this case, the addition of pectin contributed to an increase in water flux. A lower pectin concentration of 0.1 wt% resulted in a higher water flux, compared with concentrations of 0.5 wt% and 2.5 wt%.

This suggests that increasing pectin as the carbon template produces smaller pores and leads to densification. However, with only a small pectin concentration, membrane performance is more robust for sea-water desalination than when just pure silica is employed.

An advantage of using pectin as the carbon source is that it has the potential to reduce the cost of membrane fabrication and, obviously, contribute to a sustainable environment.

Acknowledgement

Muthia Elma gratefully thanks Post-Doctoral Grant 2017–2018, University Applied Research Grant 2019–2020 and Master's Thesis Research Grant 2019; Directorate of Research and Community Service; the Ministry of Research, Technology and Higher Education Republic of Indonesia and Australian Indonesian Project (ANU); Indonesian Project and SMERU Research Institute Research Grants 2016–2017; and the Australian Alumni Grant Scheme Round 2 in 2018.

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RESEARCH TRENDS

Asymmetric hydrogel–composite membranes with a self-healing capability

Hydrogel-composite materials fabricated using poly(2-acrylamido-2-methyl-1-propanesulfonic acid) (PAMPS) hydrogel and a polyethersulfone (PES) support, potentially provide a way of developing self-healing membranes – membranes that autonomously heal from physical damage without any external intervention. Previously developed pore-filled structures, however, suffer from low water permeability, since the hydrogel spans the depth of the PES substrate. This study explores a new asymmetric architecture in which the hydrogel layer is asymmetrically confined to the top several tens of micrometres of a single side of the substrate. The authors present a way of achieving this by controlling the viscosity of the acrylamido-2-methylpropanesulfonic acid (AMPS) monomer solution used during membrane fabrication. The asymmetric PAMPS composite membranes exhibited improved water permeability, compared with their pore-filled counterparts, and nearly equal self-healing performance (following the same mechanism of self-healing). The results of this study collectively suggest that asymmetric hydrogel composite membranes offer a more promising route to developing practical water filtration membranes with a self-healing capability.

B.A. Getachew, W. Guo, M. Zhong and J.-H. Kim: *Journal of Membrane Science* **578** 196–202 (15 May 2019).
https://doi.org/10.1016/j.memsci.2019.01.022

Enhanced desalination performance of PVA/carbon nanotube composite pervaporation membranes

The dispersion and interfacial interactions between nano-filler and polymer are two crucial factors affecting the performance of mixed matrix membranes (MMMs). In this study, MMMs, based on poly (vinyl alcohol) (PVA), contain-

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Submission date: 22-Jul-2020 07:39AM (UTC+0700)
Submission ID: 1360591861
File name: rampun2019_2.pdf (1.36M)
Word count: 4221
Character count: 22376

Interlayer-free silica–pectin membrane for sea-water desalination

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Water scarcity has become a serious problem worldwide. Research is now looking at the fabrication and use of interlayer-free silica–pectin membranes for sea-water desalination as one potential solution to this problem. This research work investigates the performance of interlayer-free silica–pectin membranes for artificial sea-water desalination. A dual acid–base catalyst process is applied during the sol–gel process used to prepare the membrane's thin layer structure. Membranes were prepared using tetraethyl orthosilicate as the silica precursor and various concentrations of pectin (0 wt%, 0.1 wt%, 0.5 wt% and 2.5 wt%) were used as the "templating" agent. The performance of membranes was evaluated using pervaporation (PV) processes at room temperature (~25°C). The results show that water flux for 0.1 wt% pectin was the highest (4.50 kg m^{-2} h^{-1} and 5.73 kg m^{-2} h^{-1}, at calcination temperatures of 300°C and 400°C, respectively). The fluxes at 0.5 wt% and 2.5 wt% pectin concentration were much lower, compared with 0.1 wt% (3.12–0.51 kg m^{-2} h^{-1} for both, at calcination temperatures of 300°C and 400°C). In addition, the salt rejection for all membranes remained excellent, at ~99.9%. This clearly demonstrates that carbon chains in silica matrices may produce smaller and stronger pores.

The water crisis afflicting many counties is one of the most serious problems facing communities worldwide. As populations throughout the world continue to grow this problem becomes more pressing.

To help find a solution to this problem, new approaches need to be developed to satisfy the increasing demand for clean water. Vast amounts of sea water are seen as a potential water resource.

Recently, membrane desalination using pervaporation (PV) has become a most favourable technique to produce potable water. Many pervaporative desalination studies have been done and appear in the literature.[1–5] PV has also been used to treat various salt concentrations without the need to set the pressure difference.[6]

Reverse osmosis (RO) requires the use of a high-pressure pump to treat a high salt concentration.[7]

In PV, the "driving force" behind the membrane is the chemical potential gradient between the feed and permeate. It is obtained from the difference in the partial pressures of the components on the two sides of the membrane.[8] PV has several advantages, such as a high rejection, a low operating cost and only requires low pressure.[9]

Silica

There is growing interest in using silica-based membranes for pervaporation. This is because during operation they are thermally stable.[10]

Unfortunately, obstacles to using silica membranes relate to their low hydro-stability[11] when in contact with water molecules. The concentration of silanol (Si–OH) groups in these membranes[12] also affects their properties.

Silanol is hydrophilic and when exposed to water will result in an enlargement in the pore size. It may affect membrane performance.[13]

Surface and pore silica can be easily controlled by using the sol–gel method.[14] It is also possible to modify silica and increase stability with a carbon template,[13, 15 & 16] through hybrid organic–inorganic structures[17–19] and using a metal oxide.[20–23]

Silica–carbon membranes have been fabricated successfully using the triblock copolymer Pluorionic® P123 as the template. Results show that water flux of 2.5 kg m^{-2} h^{-1} was achieved for sea-water desalination.[16] However, the use of carbon from the P123 material brings with it relatively high costs.

On other hand, a sustainable carbon precursor such as pectin has good properties that enable it to form gel.[24] In addition, it is inexpensive and an environment-friendly material. It can be obtained from orange peel,[25] apple peel[26] and banana peel.[27]

The downside is that pectin films have poor thermal stability and mechanical properties.[28 & 29] Therefore, it was selected as a template along with silica.

"Interlayer-free"

Lately, new membrane structures without interlayers have been developed. Referred to as "Interlayer-free", they are created by dip-coating sols directly onto an alumina substrate.[16 & 20]

Other work has covered generally fabricating membranes that comprise a support, interlayers and thin film in multilayers. By disregarding the interlayers the thickness of the membrane can be reduced and its performance may be improved.

For instance, the water flux and salt rejection of pure silica membranes fabricated without and with an interlayer are (8.5 kg m^{-2} h^{-1}; > 99.5%) and (2.3 kg m^{-2} h^{-1}; 91%), respectively.[13 & 16]

In addition, when comparing work that involves conventional thermal processing (CTP) and rapid thermal processing (RTP), the fabrication of membranes using CTP takes longer (in excess of one week). This is because of the slow calcination process and the need to allow 1°C min^{-1} for dwelling time.

Producing membranes using RTP takes a short period – less than one day of production time.[30] For this reason, in this work, we employ the RTP method to produce membranes.

In addition, we also use pectin as a sustainable carbon material instead of synthetic carbon,[31] and focus on investigating the performance of interlayer-free silica–pectin membranes for artificial sea-water desalination.

Experimental work

Chemicals and materials

The experimental work made use of the following materials:

- tetraethyl orthosilicate (TEOS), 99.0% (Sigma-Aldrich);
- pectin from apple (Sigma-Aldrich);
- ethanol (EtOH), 99%;
- aquadest;
- dilute nitric acid (HNO_3), 0.0008 M (Merck);
- ammonia (NH_3), 0.0003 M (Merck);
- glycerol, 85% (Merck);
- a tubular membrane support, Al_2O_3 (Ceramic Oxide Fabricators, Australia), with a length of 50 mm and outside diameter of 8 mm;
- 3.5 wt% NaCl as the feed solution; and liquid nitrogen.

Fabrication and characterisation

Sol–gel was prepared using dual acid–base catalysts by adding TEOS, drop-wise to the ethanol (EtOH, 99%), whilst stirring the solution for 5 min at a temperature of 0°C. HNO_3 was then added to the solution and refluxed for 1 h at 50°C.

This process was followed by adding NH_3, dissolved in ethanol, and the solution was stirred for another 2 h under the same conditions. The pH of the final sols was checked (pH 6).

Pectin (0.1 wt%) was dissolved into 5 ml of glycerol whilst stirring at 360 rpm for 45 minute at 40°C. This mixture was added to the pure silica sol and again stirred at 360 rpm for 45 minute at 0°C. The sol was dried in oven at 60°C to produce xerogels. After that, the xerogels were calcined in air at 300°C and 400°C.

The initial molar ratios of silica–pectin – TEOS : EtOH : HNO_3 : H_2O : NH_3 : pectin – were calculated to be 1:38 : 0.0008 : 5 : 0.00 03 : 0.000026.

Membrane preparation and pervaporation testing

Membranes were prepared via dip-coating.

The tubular support (α-Al_2O_3, with an average pore size 100 nm) was dip-coated four times using the prepared silica–pectin sol. It was calcined in air at 300°C and 400°C for 1 h using RTP. The morphology was observed using scanning electron microscopy (Zeiss), looking at a cross-sectional area and the surface of the membrane.

Membrane testing was accomplished using the pervaporation set-up that is shown in **Figure 1**.

Sea water (3.5 wt%) was prepared and used as the feed solution at room temperature (25 ± 2°C). The feed solution was vaporised and passed through the membrane. The vacuum pump (operating at ~1 bar pressure) was used to maximise the driving force.

Finally, the vaporous permeate was condensed, using liquid nitrogen, to form a liquid. The feed solution was stirred to avoid concentration polarisation. The water flux F (kg m^{-2} h^{-1}) was calculated using **Equation 1**.

$$F = [m/(A \times \Delta t)]\% \quad (1)$$

where m is mass of permeate (kg) retained in the cold trap; A is the active surface area of membrane (m^2); and Δt is the collection time (h).

The salt rejection R (%) was calculated using **Equation 2**.

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

where C_f and C_p are the feed and permeate salt concentration, which were determined using a conductivity meter SF300C-G (Ohaus).

Results and discussion

Silica–pectin membrane morphology

Membrane morphology was determined using scanning electron microscopy (**Figure 2a** and **Figure 2b**) in order to gain further understanding of the effect of morphology on membrane performance. The thickness of the interlayer-free silica–pectin membrane was around 916.3 nm.

This membrane is quite thick because of the rapid calcination process that was used. Silica–carbon sols penetrated the alumina support during the dip-coating and calcination processes.[16] This was driven by the high capillary forces present during the repeated application of the coating (four-layers) and non-uniform thickness of the thin film was observed throughout the membrane.

However, no cracks were found on the surface of silica–pectin thin film (Figure 2b).

The presence of pectin could be increasing the sol viscosity when the infiltration process is taking place. Therefore, the interlayer-free structure produces stronger and thicker pores. This is also confirmed by the excellent salt rejection (99%). In addition, as shown in our previous work, RTP results in a thicker film, compared with the CTP (470 nm).[32] The

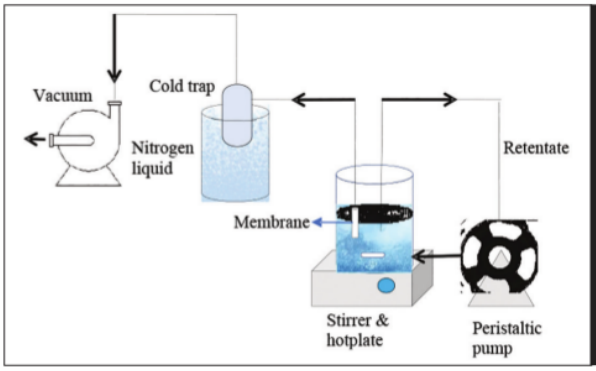

Figure 1. Pervaporation set-up for sea-water desalination.

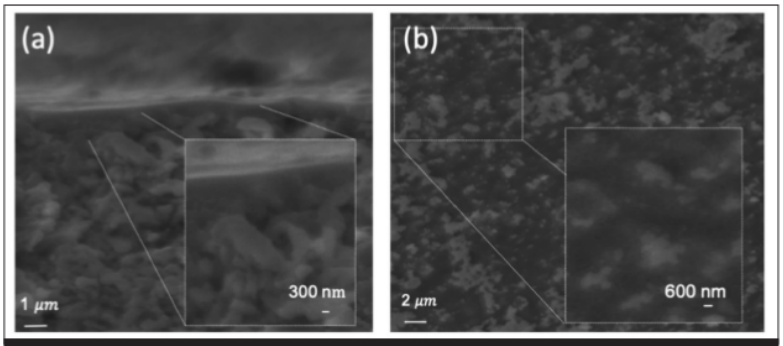

Figure 2. Scanning electron microscopy of an interlayer-free silica–pectin membrane during calcination in air: cross-sectional area (a) and surface area (b).

absence of fast calcination and ramping rate in RTP results in imperfect solvent evaporation.

Membrane performance

Figure 3 shows the performance of the interlayer-free silica–pectin membrane using artificial sea water (3.5 wt%) as the feed solution.

As can be seen, this work demonstrates that it is possible to achieve a high water flux (5.73 kg m^{-2} h^{-1}) and excellent salt rejection (> 99.9%). It clearly shows the viability of the silica–carbon membrane which uses pectin as the carbon source.

Furthermore, the promising carbon-derived material enhances silica hydro-stability for seawater desalination. Indeed, even with a low concentration of pectin (0.1 wt%) excellent performance is still achievable. This finding has the potential to reduce the cost of membrane fabrication by using a small quantity of materials.

This work has addressed the effect of pectin concentration on the silica matrix (**Figure 4**). Firstly, water flux increases sharply when pectin is used as template in the silica material (0.1 wt%).

However, when the pectin concentration is increased further, performance and water flux appear to decline. This may be because larger amounts of pectin, as the carbon concentration, can lead to membrane densification.

This means that in this case there is a limit to the increase in pectin concentration. The carbon number for a higher pectin concentration will result in the formation of non-ionic micelles, which blend with the silica network.[16]

Contrary to this, our previous study (done by Elma *et al.*[16]) stated that increasing carbon content would result in the formation of a mesoporous structure (2–50 nm). Based on that, we assume that the number of carbon chains in the pectin is much lower than that in Pluronic P123. However, the salt rejection for all of the membranes remains constant at > 99%.

In addition, Figure 3 shows the effect of temperature when RTP is used. Increasing the calcination temperature does not have much impact on water flux for a higher pectin concentration (0.5 wt% and 2.5 wt%). But interestingly, it delivered a high water flux on a lower pectin concentration (0.1 wt%) and great salt rejection.

For this reason, it is suggested that volumetric strain rises when the temperature increases. This opinion totally agrees with a previous study,[33] though other studies have also found that RTP produces a more non-porous membrane than CTP.[34] Despite the latter the use of RTP is preferred in order to reduce fabrication time.

The flux achieved in this research competes relatively well with that reported in other work, as shown by the studies listed and summarised in **Table 1.** So far, the three highest water flux

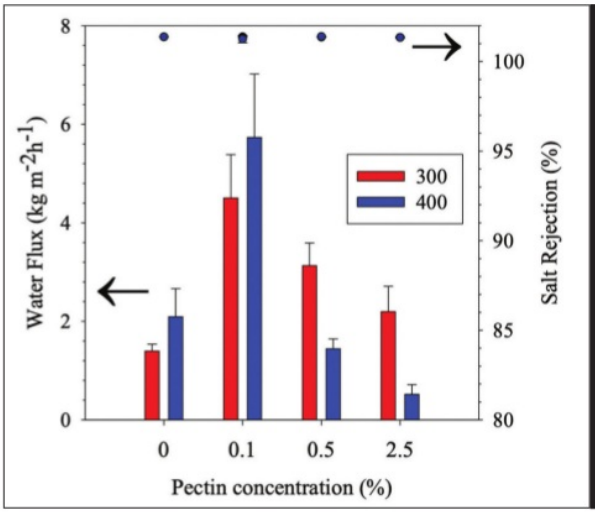

Figure 3. Effect of pectin concentration and calcination temperature on membrane performance for sea-water desalination.

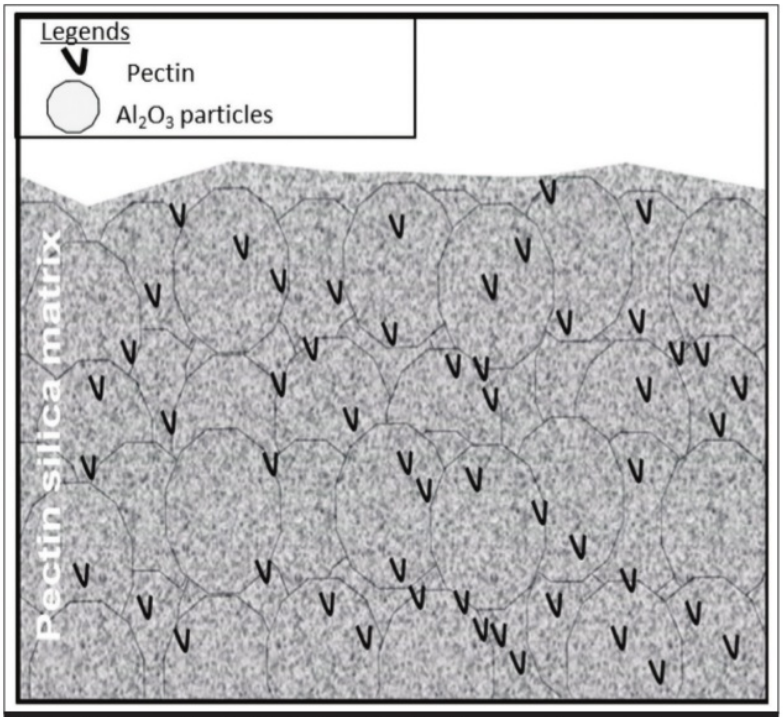

Figure 4. Illustration of pectin (0.1 wt.%) "attached" to silica on a membrane support.

figures – 20.00 kg m^{-2} h^{-1}, 19.3 kg m^{-2} h^{-1} and 17.8 kg m^{-2} h^{-1} – were achieved by Song *et.al.*, Yang *et. al.* and Wang *et.al.*

As the table reveals, different types of membranes, such as carbon–alumina mixed matrix, were used. In addition, a different precursor, ethyl silicate (ES40), was also used and all of them employed RTP. This led to greater water loss and solvent evaporation inside the xerogel because of the higher calcination temperature and formation of siloxane bridges.[33]

Besides that, our work produced high values, conducted under a feed temperature of 25°C (Table 1).[13, 16 & 35–37] Here, the acid–base catalysts are playing an important role in controlling the pore size of the membrane. Acid catalysts form small pores, whereas base catalysts promote a mesoporous structure.[38]

Small pores are certainly able to achieve a high rejection, but result in a lower flux and, vice versa, the mesoporous structure produces lower salt rejection and higher flux values.

Taking this into consideration, it is a good idea to work with a combination of a small and mesoporous construct by employing acid and base catalysts, to create a mix of a small and

Membrane type	Calcination technique	Feed temperature (°C)	Feed concentration (%)	Water flux (kg m^{-2} h^{-1})	Rejection (%)	Reference
Silica–pectin	RTP	25	3.5	5.73	> 99	(this work)
Carbon template silica	CTP	22	3.5	2.2	> 99	[16]
Carbon alumina mixed matrix	RTP	25	3.5	20	> 99	[40]
Carbon silica membrane	RTP	60	3.5	19.3	99	[17]
Ethyl silicate (ES40) membrane	RTP	60	3.5	17.8	> 99	[33]
Triblock copolymer templated silica membrane	CTP	25	3.5	3.7	98.5	[35]
Molecular sieve silica (CTMSS)	CTP	25	3.5	1.9	98	[36]
Mesoporous silica	CTP	22	3.5	6.8	> 98	[32]
Silica–TEVS membrane	N/A	22	3.5	< 2	> 99	[37]

Table 1. Comparison of silica–carbon membrane performance using rapid thermal processing (RTP) and conventional thermal processing (CTP), for desalting sea water.

mesoporous structure, referred to as a "bottle neck".[32]

In principle, water passes through membrane, but the combined pores avoid Na^+ and Cl^- release with the permeate, which have larger molecules. But this still offers a high water flux because of the mesoporous component itself.

In addition, our recent study using a single catalyst (for example, citric acid) to form a mesoporous structure, has been successful.[39]

Conclusion

In this study we have demonstrated interlayer-free silica–pectin fabrication and successfully delivered high water fluxes (4.50 kg m^{-2} h^{-1} and 5.73 kg m^{-2} h^{-1}, for materials calcined at 300°C and 400°C, respectively).

This is a significant achievement because pectin plays an important role in creating stronger pores, as evidenced by the salt rejection which remained at 99%.

In this case, the addition of pectin contributed to an increase in water flux. A lower pectin concentration of 0.1 wt% resulted in a higher water flux, compared with concentrations of 0.5 wt% and 2.5 wt%.

This suggests that increasing pectin as the carbon template produces smaller pores and leads to densification. However, with only a small pectin concentration, membrane performance is more robust for sea-water desalination than when just pure silica is employed.

An advantage of using pectin as the carbon source is that it has the potential to reduce the cost of membrane fabrication and, obviously, contribute to a sustainable environment.

Acknowledgement

Muthia Elma gratefully thanks Post-Doctoral Grant 2017–2018, University Applied Research Grant 2019–2020 and Master's Thesis Research Grant 2019; Directorate of Research and Community Service; the Ministry of Research, Technology and Higher Education Republic of Indonesia and Australian Indonesian Project (ANU); Indonesian Project and SMERU Research Institute Research Grants 2016–2017; and the Australian Alumni Grant Scheme Round 2 in 2018.

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RESEARCH TRENDS

Asymmetric hydrogel–composite membranes with a self-healing capability

Hydrogel-composite materials fabricated using poly(2-acrylamido-2-methyl-1-propanesulfonic acid) (PAMPS) hydrogel and a polyethersulfone (PES) support, potentially provide a way of developing self-healing membranes – membranes that autonomously heal from physical damage without any external intervention. Previously developed pore-filled structures, however, suffer from low water permeability, since the hydrogel spans the depth of the PES substrate. This study explores a new asymmetric architecture in which the hydrogel layer is asymmetrically confined to the top several tens of micrometres of a single side of the substrate. The authors present a way of achieving this by controlling the viscosity of the acrylamido-2-methylpropanesulfonic acid (AMPS) monomer solution used during membrane fabrication. The asymmetric PAMPS composite membranes exhibited improved water permeability, compared with their pore-filled counterparts, and nearly equal self-healing performance (following the same mechanism of self-healing). The results of this study collectively suggest that asymmetric hydrogel composite membranes offer a more promising route to developing practical water filtration membranes with a self-healing capability.

B.A. Getachew, W. Guo, M. Zhong and J.-H. Kim: *Journal of Membrane Science* **578** 196–202 (15 May 2019).
https://doi.org/10.1016/j.memsci.2019.01.022

Enhanced desalination performance of PVA/carbon nanotube composite pervaporation membranes

The dispersion and interfacial interactions between nano-filler and polymer are two crucial factors affecting the performance of mixed matrix membranes (MMMs). In this study, MMMs, based on poly (vinyl alcohol) (PVA), contain-

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