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ENHANCED WATER FLUXES STABILISATION PERFORMANCE OF TRIBLOCK COPOLYMER INCORPORATED SILICA MEMBRANES A BREAKTHROUGH APPROACH FOR NATURAL WETLAND SALINE WATER PERVAPORATION

ELMA M.[1,2,3*], RAHMA A.[2,3], GHANI R.A.[1,2], BILAD M.R.[4], ARIFIN Y.F.[5]

[1*] Chemical Engineering Department, Faculty of Engineering Lambung Mangkurat University, Banjarbaru, South Kalimantan, 70714, Indonesia
[2] Materials and Membranes Research Group (M^2ReG), Banjarbaru, South Kalimantan, 70714, Indonesia
[3] Environmental Science Doctoral Program, Postgraduate Program, Lambung Mangkurat University, Banjarmasin, South Kalimantan, 70123, Indonesia
[4] Faculty of Integrated Technologies, Universiti Brunei Darussalam, Jalan Tungku Link, Gadong, BE1410, Brunei
[5] Civil Engineering Department, Faculty of Engineering Lambung Mangkurat University, Banjarbaru, South Kalimantan, 70714, Indonesia

(*) melma@ulm.ac.id

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ABSTRACT

This work shows the preparation and water fluxes stabilisation assessment of triblock copolymer incorporated silica membrane for desalting natural wetland saline water via pervaporation. High quality triblock copolymer incorporated silica (Si-triblock copolymer) membranes delivered highest water fluxes of 4500 $g.m^{-2}.h^{-1}$ and salt rejection in excess of 99%. Water fluxes stabilisation performance confirmed that the Si-triblock copolymer membranes were hydrostable with no significant water flux and salt rejection. The hydrostability was attributed to carbonised templates embedded in the silica matrix through triblock copolymer as carbon precursor, which imparted hydrophobicity and strengthened the silica matrix under hydrolytic attack. The novelty of membrane preparation method was the calcination at intermediate temperatures of 250 to 450 C for 1 hour under atmospheric air. Rapid thermal processing (RTP) is a fast process technique for fabrication of inorganic membrane. Under these conditions, the combustion of templates is reduced, thus integrating carbonised template and silica into a single matrix membrane. The RTP proved to be a facile preparation method producing carbon silica membranes in a single day, contrary to conventional thermal process with production times up to ten days.

Keywords: Natural wetland saline water, Desalination, Triblock copolymer incorporated silica membranes, Rapid thermal processing, Water fluxes stabilisation performance.

INTRODUCTION

Access to potable water is a global problem impacting public health, particularly in dry arid areas or equatorial/tropical countries such as Indonesia (Remini, 2010; Baba Hamed, 2021; Ihsan and Derosya, 2024). Effective treatment processes, integrated approach for evaluating water quality, statistical characterization, and AI-based methods (Belhadj et al., 2017; Pandey et al., 2022; Singh et al., 2022; Sahu et al., 2024), are crucial in ensuring the delivery of high-quality drinking water to consumers, as they play a pivotal role in safeguarding public health and maintaining water safety standards (Achour and Chabbi, 2014; Aroua, 2018; Achour et al., 2019; Aroua, 2023). In these regions, climate change significantly affects both the quality and availability of water resources, exacerbating existing challenges and threatening the sustainability of water supplies for local communities (Ouis, 2012; Kouassi et al., 2013; Nichane and Khelil, 2015; Haouchine et al., 2015; Ouhamdouch et al., 2016; Assemian et al., 2021; Nakou et al., 2023; Chadee et al., 2023; Mah et al., 2024). Moreover, in these arid and semi-arid regions, enhancing the availability of water resources and artificial groundwater recharge (Chibane and Ali-Rahmani, 2015; El Moukhayar et al., 2015; Gaaloul, 2015; Bemmoussat et al., 2017; Abaidia and Remini, 2020; Qureshi et al., 2024) is essential, and this can be achieved through the effective treatment and reuse of wastewater, including both industrial and domestic effluents, to ensure a sustainable water supply (Aroua, 2022; Aroua et al., 2022; Mumthaj et al., 2023; Yadav et al., 2024).

In many of these countries, seawater and natural wetland saline waters are potential water sources that require desalination to overcome potable water access limitations (Rahma et al., 2020b; Lestari et al., 2020; Remini and Amitouche, 2023a; Remini and Amitouche, 2023b). However, it is imperative to preserve the integrity of the marine environment which can be threatened by the discharge of brines and chemicals (Amitouche et al., 2017; Belkacem et al., 2017).

Wetland areas are commonly found in tropical ecosystems, though of poor drinkable water quality such as low pH, brownish colour, and high natural organic matter (NOM) (Lestari et al., 2020; Elma et al., 2022a; Benamara and Benamara, 2022). Further, seawater intrusion in wetlands, and coastal aquifers, is common, thus increasing the salt concentration of water (Morsli et al., 2017; Djabri et al., 2019; Rahma et al., 2020b). Water aquifer is flow naturally and simultaneously carrying the other materials in system, capable of affecting water quality (Ngouala et al., 2016; Meroni et al., 2021; Later and Labadi, 2024). Among many desalination technologies, inorganic membrane desalination is an interesting application for processing natural wetland saline water using pervaporation for the provision of potable water (Wang et al., 2016; Elma et al., 2020b). Inorganic-based porous membranes such as zeolites and mesoporous silica provide a stiff ceramic structure suitable for separating water from saline waters. The separation process occurs preferentially via a molecular sieving mechanism, as the smaller water molecules

(0.26 nm) permeate through the inorganic membranes whilst excluding the permeation of the larger hydrated salt ions Na+ (0.72 nm) and Cl- (0.66 nm) (Zahratunnisa et al., 2024; Ghani et al., 2024a; Ghani et al., 2024b; Elma et al., 2024; Rahma et al., 2023; Mahmud et al., 2023).

The initial development of inorganic membranes for desalination was based on silica membranes, which are ideal for tailoring micropore sizes (Wang et al., 2014; Duke et al., 2006a). By doing so, the larger molecules such as hydrated salt ions and NOMs are blocked due to pore size exclusion and membranes tend to produce high-purity potable water. Although silica microporous membranes delivered high salt rejection, these membranes were not suitable due to low water fluxes. In addition, silica membranes are unstable in the presence of water vapor due to the presence of silanol (Si-OH) groups in the silica matrix (Wijaya et al., 2009; Sumardi et al., 2021). As a result, silica membranes undergo structural changes, leading to pore size enlargement and water flux increase, and the undesirable effect of lower salt rejection. Therefore, the inorganic membrane research started focusing on sturdier structures.

An initial candidate was zeolite membranes which are crystalline structures and precise pore size control. Several zeolite structures were prepared as membranes such as ultra-thin zeolite membranes (Yu et al., 2022). These membranes were tested for desalination reaching high water fluxes of 24-28 kg m^{-2} h^{-1} at high temperatures 50-90°C, however, the membrane stability is low due to dissolved and cracking during the pervaporation test. Back to the drawing board, researchers started stabilizing the silica structures with metal oxides (Lin et al., 2012) and carbon templates, thus improving the silica membrane hydrostability. Of particular attention, Duke et al. (2006a) reported that carbonized surfactants as templates in silica structures tend to limit the re-structuring of mobile silicas. Carbon templating of silica structures is particularly facile and desirable in the preparation of silica membranes. These pioneering works were quickly adapted for the modification of silica membranes for desalination. Wijaya et al. (2009) reported the development of carbon template from various non-ligand carbon chain surfactants C6, C12 and C16. By increasing the carbon chain, the water flux increased. Other organic materials were also used as templates such as Pectin from apple peel to prepare silica-pectin membrane for the desalination of brackish water (Pratiwi et al., 2019). Other previous work also demonstrated banana pectin template silica membrane for wetland saline water treatment by disk flat configuration and conducted high salt rejection (Elma et al., 2022b; Rahma et al., 2022a).

High quality silica membranes are generally prepared by coating two interlayers and three or four silica layers on top of a porous ceramic substrate. These membranes are calcined very slowly to avoid the cracking of the thin silica films, and it may take from seven to ten days to produce a membrane in a method called CTP (conventional thermal process). Therefore, the production of CTP silica membranes implies in high cost (Schillo et al., 2010). In order to reduce fabrication time, the sol-gel synthesis was modified allowing for silica thin film formation directly on the porous substrate (Nawi et al., 2022; Elma et al., 2021; Elma et al., 2020a), thus dispensing the need of two interlayers and cost reduction. Subsequently, Wang et al. (Wang et al., 2017) pioneered the production of high quality silica membranes in a single day only, by developing a new method RTP (rapid thermal processing). This development was possible by changing the silica precursor from TEOS to ethyl silicate E40 (Wang et al., 2017). In the RTP method, the freshly coated silica membrane is introduced directly into a pre-heated furnace for a set period thus allowing the coating and calcination of several layers in a single day. Nevertheless, there are no RTP carbon silica membranes reported in literature possibly due to the difficulties of maintaining an inert atmosphere during the introduction of the membrane into the furnace.

While the RTP method offers a lower-temperature for membrane preparation, its performance and scalability for long-term desalination application remain underexplored. Specifically, the limited operational testing duration (440 hours) may not fully capture potential membrane degradation or fouling over extended use. Additionally, the impact of air presence during RTP on membrane uniformity and defect formation has not been comprehensively studied.

In this work, we show a facile RTP method for preparing carbon silica membranes for desalination application. Instead of calcining the membranes at high temperatures of 500 to 600 °C as it is the case of CTP method under inert atmosphere or vacuum, it is postulated that a high degree of carbon template is kept embedded in the silica matrix by using the RTP method at intermediate temperatures from 250 to 450 °C for a short period of 1 h only in the presence of air. A series of RTP triblock copolymer incorporated silica (Si-triblock copolymer) membranes were developed, fully characterised, and tested for the desalination of natural wetland saline water. Subsequently, the hydrostability of the Si-triblock copolymer membranes were evaluated for 440 h in a pervaporation set up.

EXPERIMENTAL SETUP

Material and Chemicals

All chemicals were used as received. The reagents employed consisted of tetraethyl orthosilicate (TEOS, 99,0%, Sigma-Aldrich) as silica precursor, triblock copolymer pluronic P123 (PEG, 35 wt.%, Sigma-Aldrich) as carbon template, ethanol (EtOH, 97.0%) and demineralized water were used as solvent. Further, both dilute nitric acid (0.0008 M HNO_3, Merck) and ammonia (0.0003 M NH_3, Merck) were used as the dual catalysts.

Fabrication of Sol, Xerogel and Membrane

The synthesis of the silica sol procedure was detailed elsewhere (Elma et al., 2018a). Briefly, an initial silica sol was mixed TEOS and ethanol at 0 °C for 5 min, followed by the drop-wise HNO_3. Afterward, the solution was mixed and refluxed at 50 °C for 1 h. Base catalyzed ethanol-diluted NH_3 was added and mixed for another 2 h until resulting in pH 6. Final step, triblock copolymer P123 was added and further mixed at room temperature for 45 min. The final molar ratio TEOS:EtOH:HNO_3:H_2O:NH_3:triblock copolymer P123 were 1:38:0.0008:5:0.0003:0.00024. The Si-triblock copolymer sol was initially dried in an oven for a day at 60°C to form xerogels. Subsequently, the xerogels were grounded into powder and calcined in a pre-heated furnace at desired temperatures between 300–450 °C under atmospheric air for 1 h following by cooling outside the furnace until reaching room temperature.

The xerogels were characterized by TGA (Thermogravimetric Analysis) to study the mass evolution during the RTP calcination. The calcined RTP xerogels were analysed by FTIR (Fourier Transform Infrared) at wavenumber range of 400–4000 cm^{-1} (Bruker Alpha FT-IR) The structure of the RTP xerogel samples was analysed by nitrogen sorption using a Micromeritic TriStar 3020 instrument. The BET surface area and pore volumes were determined from the sorption isotherms. X-Ray Diffraction (XRD) patterns from the RTP xerogels was carried out using a Shimadzu XRD-7000.

Characterisation and pervaporation assessment of Si-Triblock copolymer Membrane

Macroporous alumina substrates α-Al_2O_3 of tubular geometry (Ceramic Oxide Fabricators, Australia) with an average pore size of 100 nm was used as membrane support. Thin films of carbon-templated silica sols were coated on the outer shell of the membrane support by a dip-coating method where the porous alumina tube was inserted in the gel using an entry and withdraw rate of 5 cm min^{-1}, and a holding time of 2 min. A total of four layers were coated and each layer times to produce 4 coating layers. Each layer was RTP calcined separately using the analogous RTP method as that for the RTP xerogels. The membrane features were examined using scanning electron microscopy (SEM, ZEISS Evo LS15) at a 20 kV acceleration voltage.

Saline wetland water (3.2 wt.% NaCl) was taken from Muara Halayung village, South Kalimantan, Indonesia at GPS coordinates of -3.5020748, 114.5727706. Fig. 1 shows a schematic of the desalination experimental setup employed for this work. The RTP Si-triblock copolymer membrane was submerged in saline wetland water and tested at varied feed temperatures at ~25 (room temperature) to 40 and 60 °C. A hotplate with a thermometer was used to control the temperature of the feed water.

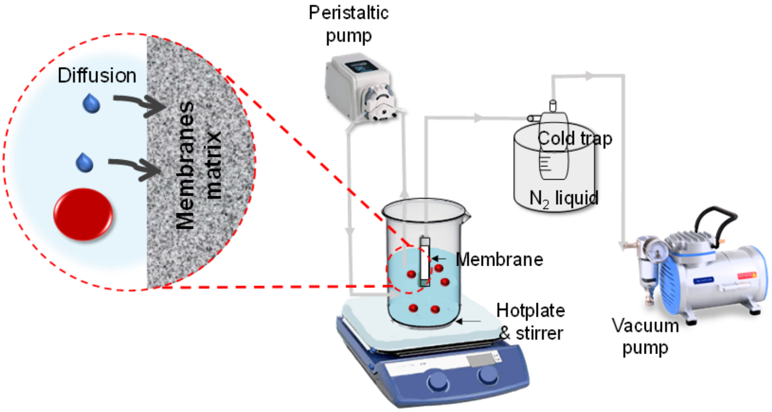

Figure 1: Schematic of pervaporation experimental setup

The water flux (F, kg m^{-2} h^{-1}) was determined according to Eq. 1, where V is the volume (L) converted from a permeate mass (kg) collected in the cold trap, A the surface area (m^2) of membrane and Δt time operation (h). The salt rejection R (%) was calculated using Eq. 2, where C_f and C_p are the feed and permeate concentrations of salt (wt.%), respectively. The salt concentrations were correlated to retentate and permeate solutions' conductivities determined by a conductivity meter (OHAUS).

$$F = \frac{V}{A\Delta t} \tag{1}$$

$$R = \frac{C_f - C_p}{C_f} \times 100\% \tag{2}$$

RESULTS AND DISCUSSIONS

As indicated in the literature (Brinker and Mukherjee, 1981), xerogels and membrane thin films demonstrate comparable behaviour regarding variations in volume and surface area. Nevertheless, the application of certain characterization techniques to membrane thin films is often challenging or unfeasible. To address this limitation, xerogels were employed as a substitute, facilitating the use of bulk characterization methods.

Xerogels Characterisation

Effect of The Calcination Temperature

Fig. 2a displays the TGA curves for a Si-triblock copolymer non-calcined xerogel. The TGA profile of Si-triblock copolymer xerogel exhibits 7% weight loss at thermal exposure before 100 °C is related to ethanol and water evaporation. From 230 to 420 °C, the weight loss of 20 wt.% is mainly associated with the decomposition of the template triblock copolymer P123 as it reacts with oxygen from air to form CO_2 that exits the xerogel as gas. After 420 °C, weight losses are minor and occur due to continuous condensation reaction of the silica matrix. Fig. 2a shows DSC of Si-triblock copolymer, the endothermic peak occurred at 56 °C and 348 °C. This peak indicates evaporation of water content and volatile compounds. Nevertheless, the glass transition cannot be observed in Si-triblock copolymer xerogel sample due to there is a disturbance in the crystalline region (Wu et al., 2014). In other hand, the melting peak of the Si-triblock copolymer xerogel was appeared at 244 °C and 349 °C because of thermal decomposition of carbon from P123 degraded partially. Meanwhile, the exothermic peak on the Si-triblock copolymer xerogel emerged at 374 °C, where the peak indicates that membrane have decomposed. Interestingly the thermal resistance of the Si-triblock copolymer membrane is better than another silica-based membrane with cellulose acetate addition as primer material at 421 °C. The amorphous carbon is associated with the carbon visually observed in the RTP xerogels in Fig. 2b.

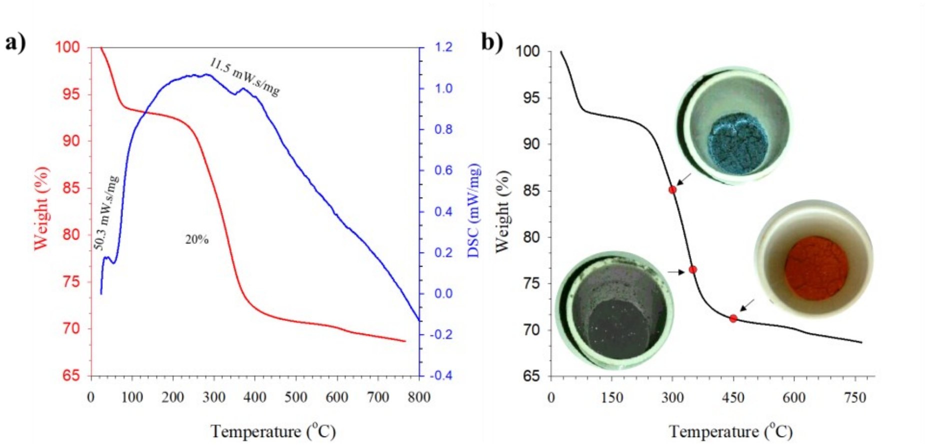

Figure 2: a) Thermogravimetric weight loss curves of Si-triblock copolymer xerogels calcined at 350 °C, b) images of xerogels calcined at various temperatures.

Fig. 2b depicts images of RTP xerogel samples calcined at various temperatures. It is observed a visual evolution whereas at lower temperatures (300 and 350 °C) the xerogels are characterised by dark black colour and at the higher temperature (450 °C) red colour dominates with small black grains. These results are interesting and are contrary to conventional air calcination where silica carbon xerogels exhibit white colour due to the combustion of carbon templates. Although the RTP xerogels were calcined in atmospheric air as well, the fast heat treatment afforded by the RTP method gives a clear indication of air diffusion limitation to burn off the triblock copolymer P123 surfactant embedded into the silica matrix. As RTP method involves a direct air calcination for 1 hour in this work, conventional air calcination can take from 10 to 15 hours for a complete cycle of heating and cooling for the same temperatures using in this work. Therefore, the short time used in the RTP method is not enough to completely combust the triblock copolymer P123 surfactants into CO_2.

Fig. 3a shows a representative FTIR spectra in range of 700-1400 cm^{-1} for the functional groups of Si-triblock copolymer matrices derived xerogels. The FTIR bands at 802, 1070 and 1218 cm^{-1} are assigned 70 the formation of siloxane (Si-O-Si) groups (Wijaya et al., 2009). Silanol (Si-OH) bands appear at shoulder at ~962 cm^{-1} which similar to previous study investigated silica base membranes (Elma et al., 2022c; Elma et al., 2022e; Sumardi et al., 2021). The formation of silanol and siloxane groups are a direct result of the silica sol gel synthesis related to the hydrolysis and condensation reactions, respectively (Rahman et al., 2020: Pratiwi et al., 2019). The XRD patterns in Fig. 3b shows a broad peak between 15 and 34° 2θ irrespective of the calcination temperature. This indicates the presence of amorphous structures of silica and carbon which appear at the same 2θ range.

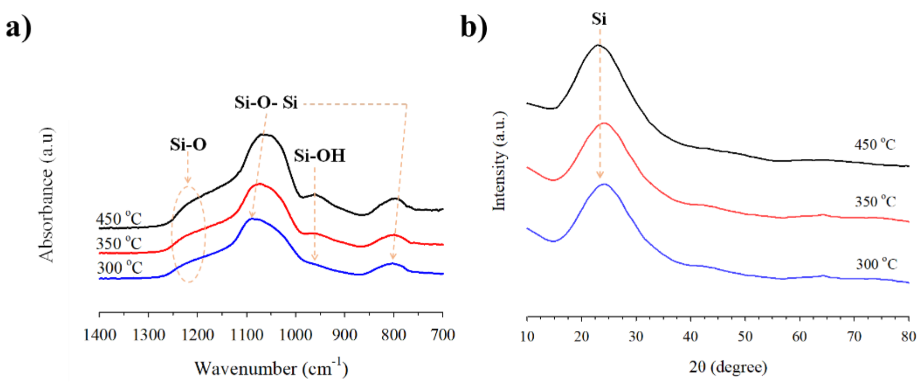

Figure 3: a) Fourier transform infrared spectra, b) XRD patterns for xerogel samples calcined at various temperatures.

Fig. 4a exhibits the N_2 sorption isotherms of the RTP xerogels calcined at various temperatures. All RTP xerogels are characterized by type IV isotherm with H4 hysteresis. These results indicate that the RTP xerogels formed micro and mesoporous structures. The microstructure is observed for the stronger sorption values for relative pressures $p/p^o < 0.25$. The mesoporous structure for all RTP xerogels is related to the formation of a hysteresis for each sorption isotherm at $\sim 0.4 < p/p^o < \sim 0.8$. Silica derived from acid base catalyst method tend to form microporous structures as reported elsewhere (Jillani et al., 2024). This formation of mesoporous structures in this work may be related to partial degradation of the triblock copolymer P123 templates as evidenced by the black color in the xerogel samples in Fig. 2.

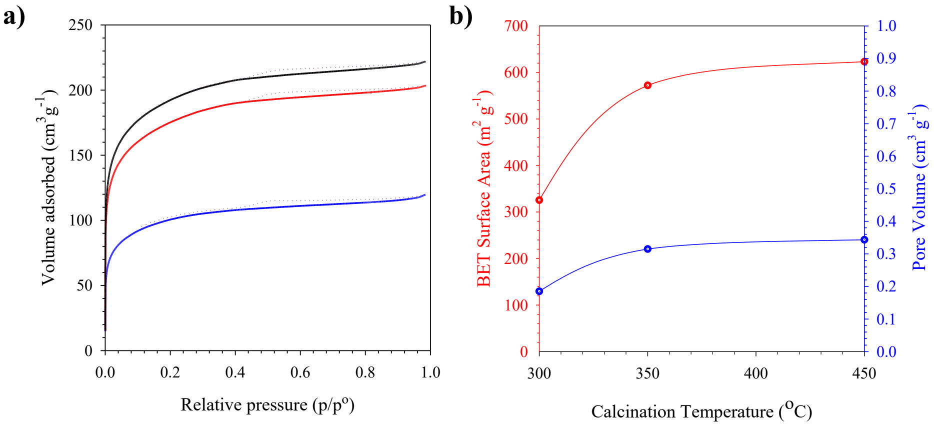

Figure 4: a) Nitrogen adsorption isotherm, b) BET surface areas and pore volumes for RTP caron silica xerogels calcined at various temperatures.

Fig. 4b displays the BET surface area and pore volumes of the RTP xerogels. It is observed a sharp increase in both parameters by almost twofold from 300 to 350 °C followed by a relative low increase to 450 °C. These results correlate well with the weight loss in Fig. 2a. For instance, the sharp weight loss between 300 and 350 °C is assigned to the degradation of triblock copolymer P123 template and an increase in pore volume. The increase in BET surface area is interesting as this parameter is mainly associated with the first layer coverage at for relative low pressures $p/p^o < 0.25$. These results suggest that a partial degradation is the prevalent mechanism, instead of full degradation that leaves a cavity. This means that the partial degradation of P123 forms carbon micro-structures within the silica matrix. As the RTP temperature increases from 350 to 450 °C, the small increase in both BET surface area and pore volume is also related to weight loss (Fig. 2a), though at this temperature range the triblock copolymer P123 weight loss is minor.

Membrane Morphology

SEM images of the cross section on Fig. 5 shows the morphology features of the RTP silica carbon membranes. The top layer thickness decreased from ~1 to ~0.90 µm as the RTP temperature increased from 300 to 450 °C. The reduction of the top layer thickness correlates well with the reduction of mass as observed in the TGA results (Fig. 2a). The top layers have rough features in line with the pioneering work of Wang and co-workers (Wang et al., 2017) on the development of RTP silica membranes. This is attributed to the fast heat treatment that stresses the thin film structure. Further, the top layers in this work were directly deposit on the substrate, dispensing of the need of smooth interlayers. Alumina substrates are rough and the thin film top layers tend to follow the substrate roughness features (Elma et al., 2022a: Rahma et al., 2019; Sari et al., 2023; Elma et al., 2023; Sumardi et al., 2021; Rahman et al., 2020; Rahma et al., 2020a).

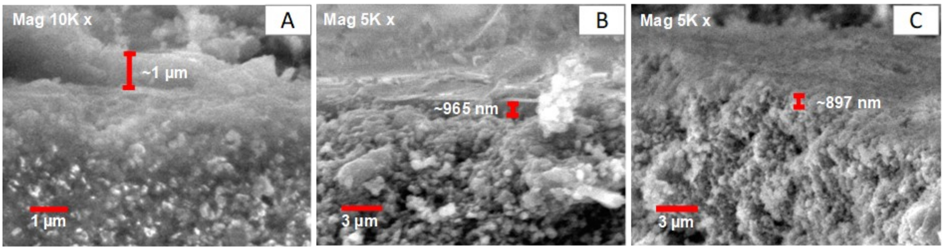

Figure 5: Scanning electron microscopy image of membranes calcined at (A), 300 (B) 350 and (C) 450 °C via the RTP processing method (cross section perspective).

Membrane Performance for Natural Wetland Water Pervaporation

Effect of The Feed Temperature

Water flux of the Si-Triblock copolymer membranes calcined at various temperatures are displayed in Fig. 6 for desalination of wetland saline water. All membranes delivered high salt rejection above 99%. The highest water fluxes of 4.5 kg m^{-2} h^{-1} at 60 °C were measured for the RTP membranes calcined at 350 °C. All water fluxes increased as a function of the feed temperature. This is attributed to an increase in the driving force, as the vapor pressure on the feed side increases due to temperature raising, while the vapor pressure on the permeate side remains constant. The raising of vapor pressure leads to an increase in the driving force, thus improving the water flux. The higher temperature makes it easier for water to passed across through the membrane, which is confirmed by the results that the diffusion coefficient of water increases by four times as the feed temperature is raised from 20 to 60 °C.

The water fluxes increased as the membrane was RTP calcined from 300 to 350 °C (Fig. 6). This trend is in line with the N_2 sorption isotherms (Fig. 4a) as an increase in pore volume reduces the resistance to water diffusion leading to an increase in water fluxes. Contrary to this trend, the water fluxes reduced for the RTP calcined from 350 to 450 °C even though pore volumes increase slightly (Fig. 4b). This unexpected result suggests a densification of the thin films. Indeed, this is the case as the SEM images (Fig. 5) shows that the membrane RTP calcined at 450 °C thin film thickness of 897 nm reduced an average of 7% as compared to that of 965 nm of membrane RTP calcined at 350 °C. These results strongly suggest that the RTP method is optimal at around 350 °C in terms of water fluxes. By the same token, higher RTP calcination of at 450 °C is undesirable due to thin film densification.

The RTP Si-triblock copolymer membrane calcined at 350 °C and pristine silica membrane calcined at 600 °C were long-term tested for 400 h for wetland saline water as displayed in Fig. 7. All water fluxes varied within the same experimental error region of 2.5 and 2.8 kg m^{-2} h^{-1} with an average value at 2.6 kg m^{-2} h^{-1} (for RTP with P123 template silica membrane). Salt rejection was high over 99% with marginal variations close to 100%. These results strongly suggest that the RTP membrane was hydrostable during this long-term testing of 400 h compared with the silica membrane without P123 templated. This is attributed to carbon embedded into the silica matrix, a method pioneered by Duke et al. (2006a), as carbon imparts hydrophobicity and maintains the integrity of the silica matrix. After 400 h testing, the RTP membrane started fouling and the experimental work ceased. Nevertheless, it is noteworthy that the RTP membrane performance was consistently stable during this water flux stabilization performance testing, particularly that wetland saline water contains organic matter (NOM molecules of 1-100 kDa (Xu and Guo, 2017: Haan and Boer, 1987)) as evidence by the water analysis in Table 1, in addition to other salts. Silica membrane by templated carbon proven to enhance life time of membrane performance for desalination as mentioned in other works (Suhendra et al., 2022: Rahma et al., 2022b: Elma et al., 2022d: Elma et al., 2022a).

Fig. 7 plots the permeate NaCl concentration in ppm and compares it against the recommendation for maximum salt allowable in potable water according to the World Health Organization WHO. The RTP membrane performed very well up to 120 h as permeate salt concentration was not significant (i.e., close to 0 ppm). After 120 h, the salt concentration increased to 80 ppm, well below the WHO standards of 600 ppm. Interestingly, the increase in salt concentration in the permeate scream was accompanied by a slight increase in water flux. This is a clear indication that the silica matrix of the membrane is re-arranging itself under hydrophilic attack. Nevertheless, these results confirm the suitability of the Si-triblock copolymer membrane for producing potable water from wetland saline water. By comparison, the conventional silica membrane starts to densify during filtration after 200 hours.

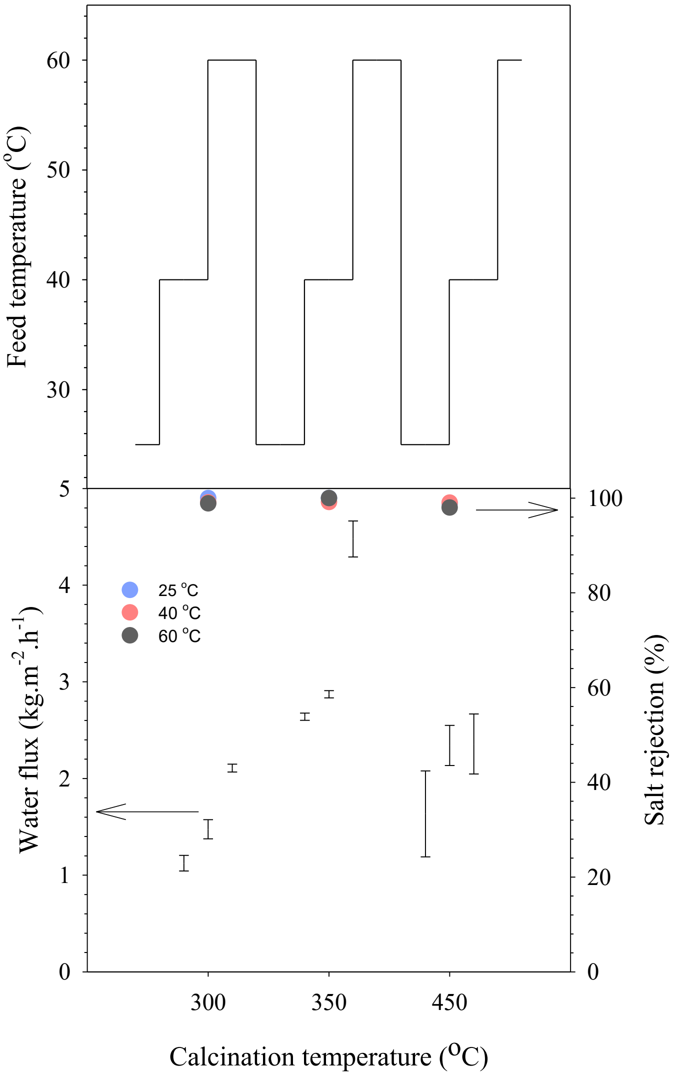

Figure 6: Membranes Silica- Carbon Pluronics ® triblock copolymers P123 performance as a function of water flux and rejection of natural wetland saline water to varied feed temperature and membranes calcined temperature.

Table 1: Characteristic of wetland saline water taken from Muara Halayung.

Parameters	Result
pH	5.7-5.8
Color	Brown Yellow
Odor	Odorless
Salinity (wt% NaCl)	3.2
TSS (ppm)	1350
TDS (ppm)	986
EC (µS/cm)	135.60
TOM (ppm)	15.20
TOC (ppm)	10.20
UV absorbance 254 (cm^{-1})	0.460
Al (ppm)	4.458
Ca (ppm)	3.671
Mg (ppm)	6.364
SO_4^{2-} (ppm)	17.90
Total Hardness - $CaCO_3$ (ppm)	35.37

Silica by itself is well known to suffer degradation under hydrophilic attack (Duke et al., 2006a). This causes pores of conventional silica membranes to undergo structural re-arrangement after 350 h testing. It led to pore size reduction as water fluxes slightly reduced around testing resulting in very low salt concentration in the permeate stream. Further, the silica membrane without embedded carbon carried out the mobile silica to rearrange itself under hydrolytic attack in line with the model proposed by (Duke et al., 2006a). Therefore, Si-triblock copolymer membrane poven has great performance due to carbon imparts hydrophibicity, thus enhancing the RTP membrane hydrostability.

Fig. 8 schematically elucidates the transport phenomena associated with Si-triblock copolymer membranes. The high salt rejection in excess of 99% (Figs. 7) clearly shows that diffusion of hydrated ions of Na and Cl of ionic diameters of 0.72 nm and 0.66 nm was hindered. Salt was measured at low levels after 150 h testing (Fig. 8a), suggesting a minor re-arrangement of the silica matrix. Therefore, these results suggest that the majority of the pores formed between silica and carbon were below of that from Na and Cl ionic diameters. In this case, the diffusion of water molecules with a kinetic diameter of 2.8 Å prevailed. After 150 h, a very small amount of percolation pathways were formed within the Si-triblock copolymer membranes, thus allowing for the diffusion of salts at very low ppm concentrations (Fig. 7). Nevertheless, the Si-triblock copolymer membrane retained its hydrostability during the 440 h long term testing when fouling took place.

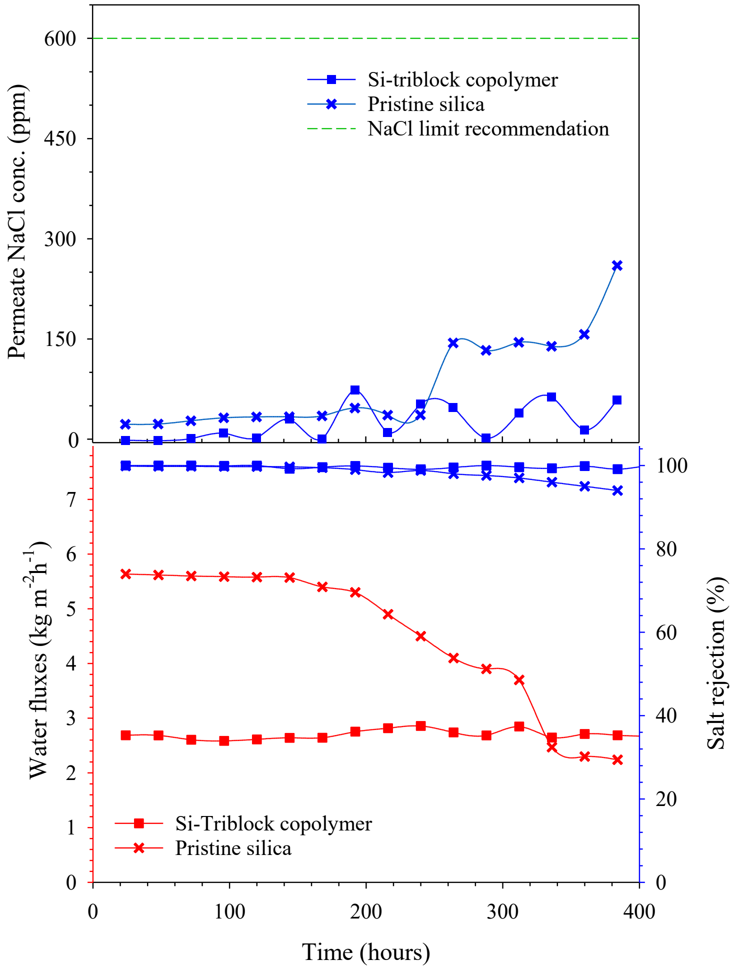

Figure 7: 400 hours long term testing of (below) water fluxes and salt rejection, and (above) permeate salt concentration of the silica-triblock copolymer membrane as a function of time. Conditions: natural wetland saline water at room temperature (25 °C).

The overall performance of the Si-triblock copolymer membranes is compared against other inorganic membranes reported in the literature for desalination at room temperature. Interestingly, Si-triblock copolymer membrane in this work more favorable to desalting of wetland saline water about 4-fold higher than previous work reported (Elma et al., 2018a: Lestari et al., 2020: Elma et al., 2018b). On the other hand, silica membranes without carbon templates performed high water fluxes but their stability performance was not worth desalination (Elma et al., 2018c). Moreover, salt rejection of Si-triblock copolymer membranes in our work resulted in good and competitive other carbon silica template membranes (Yang et al., 2017).

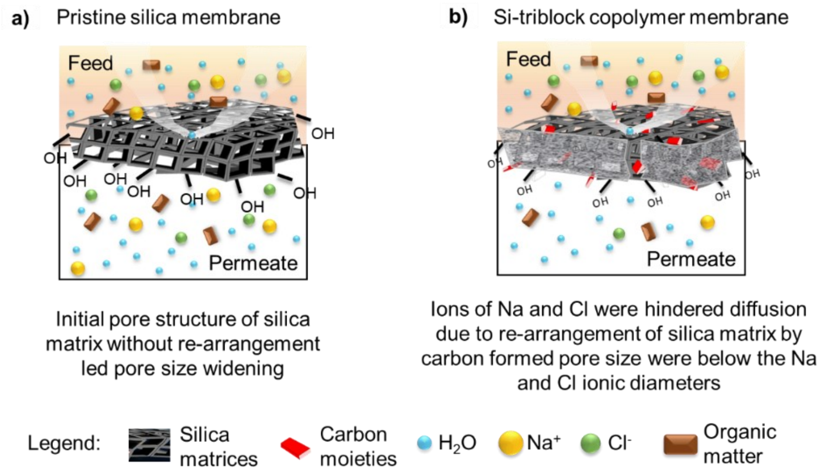

Figure 8: Idealised schematic of water diffusion through the Si-triblock copolymer membrane.

Comparison with Other Reports

A comparison of the results of this work with previous studies reported water flux performance of silica-based membranes and others, as well as data obtained using varied salt concentrations through pervaporation shown in Table 2. It can be observed that the Si-triblock copolymer membrane performed greater pervaporation efficiency than other membranes for non-artificial saline water under the same condition. In addition, the Si-triblock copolymer membrane was prepared using a rapid thermal processing technique, which brings it more efficient and low-cost production.

Table 2: Comparison of saline feed water desalination via pervaporation using silica-triblock copolymer membranes and other membranes

Membrane	Feed solution	Feed temp. (°C)	Saline feed conc. (wt %)	Water flux (kg.m-2.h-1)	Ref
Si/C16	artificial	25	3.5	3	(Wijaya et al., 2009)
Si/MTEOS	artificial	25	3.5	1.8	(Duke et al., 2007)
Si-triblock copolymer	Natural wetland saline water	25	3.2	2.6	This work
FAS (fluoroalkylsilane) grafted	artificial	40	2.9	4	(Kujawski et al., 2007)

FAS (fluoroalkylsilane) grafted	seawater	40	-	2	(Kujawski et al., 2007)
Si-triblock copolymer	Natural wetland saline water	40	3.2	2.9	This work
PVA/Si	artificial	60	0.2	20.4	(Chaudhri et al., 2018)
PVA/green Si	Raw seawater	60	2.6	7.1	(Silva et al., 2021)
PVA/green Si	Pretreated	60	2.6	10.6	(Silva et al., 2021)
Si-triblock copolymer	Natural wetland saline water	60	3.2	4.5	This work

As summarized on Table 2, it can be noted that few works reported the pervaporation process with artificial saline water, real seawater, and pretreated seawater for the membranes developed. Kujawski et al. (2007) reported a FAS (fluoroalkylsilane) grafted ceramic membranes for desalting seawater taken from the North Sea coast at Scheveningen as feed water demonstrated a lower permeate flux about 30%. Furthermore, the water flux of Si-triblock copolymer membrane at 25 °C feed temperature showed greater than previous study reported using Si/MTEOS membrane nearly double fold (Duke et al., 2007).

CONCLUSION

Carbon silica membranes ware successfully prepared by RTP method at intermediate temperatures (300-450°C) under atmospheric condition. All membranes demonstrated excellent performance of water flux and salt rejection when applied for seawater and wetland saline water desalination. Si-triblock copolymer membrane prepared and calcined at 350 °C shows the highest water flux of 4.3 $kg.m^{-2}h^{-1}$ (wetland saline water) and 3.9 $kg.m^{-2}h^{-1}$ (seawater) at feed temperature 60 °C. The surface area of this membrane exhibits high of 572 m^2g^{-1} for Si-triblock copolymer sample calcined at 350 °C. Furthermore, the structure of this membrane found mesopore (2.2 nm) that is very suitable when applied for water desalination. In addition, the salt rejection performed extremely good for over 98% for all membranes testing. Regarding the stability testing of carbon template mesoporous silica membranes, this type of membrane successfully stable for over 16 days when applied for desalination of wetland saline water. It was also observed that the membrane long-term performance achieved water flux remained stable (from 2.3 to 2.8 $kg.m^{-2}h^{-1}$). As well as salt rejection, it shows stable up to 99% and comply with WHO potable water standard. It decided that calcined carbon template mesoporous silica membrane derived P123 is robust and potential to considered for desalination of seawater and wetland saline water particularly.

Declaration of competing interest

The authors declare that they have no known competing interests or personal relationships that could have appeared to influence the work reported in this paper.

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