

Novel Multi-Channel Coated Silica Based Membranes Applied for Peat Water Ultrafiltration

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ARTICLE INFO	ABSTRACT
Article history: Received 15 May 2022 Received in revised form 11 October 2022 Accepted 23 October 2022 Available online 11 November 2022 Keywords: Peat water; pure silica; silica-pectin	South Kalimantan-Indonesia is in the Southern part of Borneo which almost half of the population lives in remote areas. Geographically, people are isolated and have poor access to electricity as well as a clean water supply. It is very essential to ensure the availability of clean water produced from peat water to overcome future water shortages. This work performs the fabrication of multi-channel silica-based membranes applied for peat water ultrafiltration which is demonstrated under various transmembrane pressure (TMP) and top layer types. The silica based top layer membranes were fabricated from three different sources (silica-pectin, organosilica, and pure silica) via dipcoating method onto macroporous multi-channel alumina support (4 bore). Peat water was treated by multi-channel membranes through ultrafiltration process through cross flow system. According to the results, all multiples top layer pore size were obtained with range from 2.1 to 6 nm classified as mesoporous membranes. This process exhibited a promising outcome for removal natural organic matter (NOM) about 89-71% (UV ₂₅₄ rejection) at TMP 1-3 bar, respectively. Besides that, normalized permeate fluxes of that membrane excellently stable over 180 min operation time. It is concluded that silica based multichannel membranes are either
membrane; organosilica; ultrafiltration; multichannel; NOM; TMP; cross flow	compatible to treat peat water that has high number of NOM as well as excellent to overcome membrane fouling.

1. Introduction

Half of the population in South Kalimantan, Indonesia lives in rural areas, which agriculture and fishery are the main source of income. They are often geographically isolated and lack access to clean water supply. Mostly rural homes may use water from river or peatland water. However, this is typical only done during a well or dry season is not available. Moreover, direct consumption of peat water is not advisable due to its characteristic rich by organic substances. There are multiple techniques to remove organic substances such as chemical process; chemical coagulation; biological

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process; such as activated sludge, and physical process; mainly filtration [1,2]. Therefore, a versatile water treatment technology like membrane separation is useful in this case [3-5].

Ultrafiltration is a membrane technology widely used as pretreatment or primer process in water purification plants [3,6-8]. This technology provides the attracting features of simple operation, less energy consumption, lower footprint, and lower cost [9-14]. Silica is an interesting material to applicates as ultrafiltration membrane, due to provide good molecular sieving, robust, resistant to high temperature and chemical, and more antifouling over polymer-based membrane [15]. Nevertheless, silica membrane also has a major drawback which has low stability when contacted with water [16].

Several strategies have been demonstrated to overcome the disadvantage of silica membrane by carbon template [17] and carbon hybrid silica membrane [18]. Carbon templated silica are considerably as an excellent strategy for membranes preparation applied for water treatment due to the hydrostability of the silica membrane is enhanced [10,13,15,19-29]. Furthermore, the hybrid carbon-silica membranes prepared from citric acid as catalyst is also produced carbon groups. This aims to strengthen the silica matrix [30,31]. The pure silica membrane applied for natural wetland water desalination is firstly reported by Elma and Assyaifi [32]. Then, other study was employed pectin from banana peels, citric acid and copolymer P123 as a carbon source which was incorporated into a silica matrix to enhance the hydrostability [24,29,33,34]. Other than that, banana peel is condsiderability for water clarification due to highly potential application as carbon carbon agent and bio-flocculant for water clarification [35]. Assyaifi et al., [36] has been also reported, pectin could increase silica membrane performance for wetland saline water desalination via photocatalyticpervaporation. The previous study have been succeed to fabricate silica-pectin membrane applied for wetland saline water desalination via pervaporation method [11,26,37-39]. Sumardi et al., [10] has been successfully demonstrated organosilica membranes fabricated by addicted citric acid as acid catalyst applicated for water desalination. All of previous studies have shown carbon-silica based membranes fabricated with single channel tubular configuration, none of them have multichannel tubular configuration.

Commercially, tubular membrane support configuration has multiple type such as single and multichannel [21,31,34,38,40-50]. Multichannel tubular membrane offers high surface area than single one. Due to that, multi-channel membrane might potentially to developed and implemented for scaled up to provide potable water among rural communities. Multichannel membranes provide the bore more than one that effected the surface area of the membrane increase and produce higher flux than single channel membrane [51]. The objectives of this work are to investigate the characterization and morphology of pure silica membranes and excellent hydrostability membranes derived from pectin and organosilica fabricated employing multichannel tubular configuration. In addition, this work also demonstrates the performance of these membranes applied for peat water treatment via ultrafiltration process.

2. Methodology

2.1 Materials

The preparation of silica-based sols and membranes were conducted using primer of chemicals and materials such as: silica precursor from TEOS-tetraethyl orthosilicate (99% purchased from Sigma-Aldrich), organic solvent ethanol (96%, distribution from local company PT. Sumber Kita Indah, Bekasi-Indonesia), and demineralized water. Citric acid was purchased from Sigma-Aldrich and nitric acid obtained from Merck as acid catalyst, while base catalyst using ammonia purchased from Merck. Pectin as carbon template agent extracted from local banana peels waste [52]. Multichannel alumina membrane support (four bores, α -Al₂O₃ tubular substrate) was purchased from Ceramic Oxide Fabricators, Bendigo-Australia. Meanwhile, peat water as feed for ultrafiltration experiment was collected from Jalan Suka Maju, Banjarbaru, South Kalimantan-Indonesia at GPS coordinate of - 3.405418, 114.719799.

2.2 Silica-Based Sols Preparation and Characterization

Preparation of silica-based sols were conducted via sol gel method. There are three types of thin film were used in this work, the composition and properties of all thin film are shown in Table 1, while the detail procedure of sols preparation can be seen in the previous work [11,15,49]. In order to characterize silica-based sols, the multiple sols were dried for overnight at 60 °C by oven, followed by grounded the dried sols to be powder. The dried silica-base sols then named as xerogel, afterward the varied silica-based xerogels were sintered under air condition at 600 °C for pure silica xerogel. In other hand, organosilica xerogels was sintered at 175 °C while silica-pectin at 300 °C under vacuum condition by furnace for an hour. These all xerogels sample were characterized by FTIR (Fourier Transform Infrared) – Bruker Alpha spectrometer at wavenumber range of 600–2000 cm⁻¹ to functionalize the chemical properties of xerogels and the deconvolution of silanol/siloxane peaks derived from FTIR data. Then these data were curved fitting using Fityk software program by Gaussian approach to determine the quantitative of peak area ratio of functional group.

Tabel 1

The silica-based thin file	n composition and	properties in this work
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	Pure silica	Organosilica	Silica-pectin
Ref	Elma <i>et al.,</i> [49]	Elma <i>et al.,</i> [15]	Mustalifah et al., [11]
Acid catalyst	Nitric acid (0.00078 N)	Citric acid (0.001 N)	Nitric acid (0.00078 N)
Base catalyst	NH₃ (0.0003 N)	NH₃ (0.0003 N)	NH₃ (0.0003 N)
Templated agent	-	-	Banana peel pectin
Calcination temp (°C)	600 (air)	175 (vacuum)	300 (vacuum)
Active surface area m ²	0.002		
Alumina substrate	Multichannel (4 bores)	Multichannel (4 bores)	Multichannel (4 bores)
configuration			

2.3 Multichannel Silica-Based Membranes Fabrication and Characterization

Fabrication of silica-based membranes were undertaken by coated the inner surface of multichannel support using silica-based thin film were have been prepared in 2.2 sub section. In this work, it has been prepared three types of membranes which coated by pure silica, organosilica and silica-pectin sols that every type membrane was producing 4 coating layers. Membrane was calcined under vacuum condition in every layer of coating by using furnace at different temperature 600, 175 and 300 °C for pure silica, organosilica and silica-pectin, respectively. The schematic of inner coating equipment of membrane fabrication displays in Figure 1. The morphology of multichannel silica-based membranes was determined by scanning electron microscopy (SEM, ZEISS).



multichannel silica-based membrane fabrication

2.4 Multichannel Silica-based Membranes Performance for Peat Water Ultrafiltration

Performance of multichannel silica-based membranes for peat water treatment processed by ultrafiltration. Schematic of laboratory-scale ultrafiltration set-up for testing peat water can be shown in Figure 2. A tubular module was placed as reactor for separation of natural organic matter that therein containing of multichannel silica-based membrane. Ultrafiltration of multichannel silica-based membranes were tested by cross flow system at different transmembrane pressure (TMP) 1-3 bar for an hour filtration time at room temperature (25 °C \pm 2 °C). To supply the pressure for filtration, the system completed with booter pump, meanwhile the permeate volume was collected every 5 min.

The permeate flux (J, $Lm^{-2}h^{-1}bar^{-1}$) was calculated according the Eq. (1), where V is a permeate volume (L), A the membrane active surface area (m²) and Δt is operation time (h). Whereas, the rejection of contaminant (R, %) was determined using Eq. (2), where Cf and Cp are the feed and permeate value of UV absorbance 254, respectively. The UV₂₅₄ parameter was measured using Spectrophotometer Genesys 10S UV-Vis.

$$J = V/(A.\Delta t)$$
(1)

R= (Cf-Cp)/Cf ×100%,

(2)



water treatment using multichannel silica-based membranes

3. Results and Discussion

3.1 Silica-Based Membranes Functionalization and Deconvolution

In this work, three type of thin film (pure silica, organosilica, silica-pectin) were generated to compare the multiple xerogels characterization for fabrication of multichannel membranes. The all variation of thin film were chosen from the optimum sample in the previous work which applied for water desalination [11,15,49]. Pure silica xerogel was interpretated the silica membrane without the addition of template agent in order to enhance the hydrostability of silica matrix. Meanwhile, the organosilica and silica-pectin xerogels were represented the addition of carbon into silica matrix by hybrid (organosilica) and template (silica-pectin) strategies for each thin film type in order to improve the silica matrix stabilization in water.

Fundamentally, the silica matrix structure has high concentration of the surface hydrophilic silanol (Si-OH) species that contributes to decrease the hydrostable of silica structure during contacted with H₂O molecules [21]. FTIR spectra was examined to investigate the presence of silanol and other species were generated from multiple xerogels of silica-based sample. Functional groups of silica-based xerogels were prepared via sol gel method exhibits on Figure 3(a) to representative of FTIR spectra in range of 600-2000 cm⁻¹. The three of xerogels sample (pure silica, organosilica, and silica-pectin) xerogels presented several functional groups that are silanol (Si-HOH), siloxane (Si-O-Si), and silica-carbon (Si-C). The lower peak of Si-C was displayed on Figure 3(a) for pure silica xerogel, due to there is no carbon addition likes other two sample for organosilica from citric acid and silica-pectin xerogels from pectin loading. Organosilica and silica-pectin xerogels resulted to generate Si-C stretching vibration at wavelength 779 and 795 cm⁻¹, respectively. The functionalization peaks of silica-carbon bands are risen because the wagging mode of the carbon attached to Si which similar result also can be found to previous study [53].

The silanol and siloxane groups were formed via reaction of hydrolysis and water and alcohol condensation during the sol-gel process. The Si-O-Si stretching linear vibration also can be encountered at 1063-1077 cm⁻¹ wavelength as either reported in previous results on silica membranes-based materials [16,17,54]. The silanol (Si-OH) peaks appear at shoulder of Si-O-Si peak near 946-966 cm⁻¹ for all varied xerogels sample as displays on Figure 3(a). Siloxane is formed by water and alcohol condensation reaction when sol-gel process. Meanwhile, the silanol was obtained

from the hydrolysis reaction at first-step process by addiction of acid catalyst (nitric acid and citric acid for preparation of (pure silica and silica-pectin) and organosilica, respectively. Addition of acid catalyst into the silica sol promoted the rate of the hydrolysis reaction [22]. The addiction of pectin and carbon derived from citric acid into silica matrices contributes to enhance the hydrostability of silica matrices that is proved by the presence of Si-O bond, where Figure 3(b) is the deconvolution peak ratio of silanol/siloxane. It is appropriated to the lates study that reported elsewhere [17]. This finding can also be explained as good indication to improve silica network and perform more stable material applied for water treatment.

Interestingly, Figure 3(b) shows the green scatter of plotting peak area ratio of Si-OH/Si-O-Si, while pink scatter appears the area of silanol or Si-C peak shown at wavelength of ~790 cm⁻¹. The lower peak area ratio of silanol over siloxane obtained for the silica-pectin xerogels, whereas the higher for pure silica xerogels sample. The lower area ratio of silanol over siloxane was indicated the presence of siloxane species dominated the silica structure compare to silanol. It can be concluded that the hydrostability of silica structure may led become excellent related to pure silica [10].



Fig. 3. (a) The Fourier-transform infrared spectroscopy spectra of silica based xerogels calcined in vacuum with their (b) area ratios between silanol (940 cm⁻¹) and siloxane (1080 cm⁻¹) in green and in pink for area of siloxane at 790 cm⁻¹

3.2 Multichannel Silica-Based Membranes Morphology Structure, BET Properties and Pure Water Permeability

Figure 4 performs the SEM images of all multichannel silica-based membranes surface area that indicates the morphology structure. The surface morphology of pure silica membrane looks smoothers than organosilica and silica-pectin membranes (Figure a, b, and c). It is due to the SEM image on Figure 4(b) and (c) containing carbon, so the surface of top layer become rougher than Figure 4(a). This result also similar to other work which employed pectin from apple as carbon template to fabricate silica-pectin membranes [55].

The outlook image of the membranes is presented in high resolution to display the effect of the coating on inner surface of the multichannel substrate. In all of Figure 4 the coated membrane of multichannel silica-based membranes can be seen the top layer have the roughness and boulders of the alumina particles substrate protruding on multiples membrane surface. Moreover, the top layer

of all membranes looks not delicate due to its membranes were coated without interlayer, or called as interlayer-free as first time reported by Elma *et al.*, [22].



Fig. 4. The scanning electron microscope images of (a) pure silica; (b) organosilica; and (c) silica-pectin multichannel membrane calcined in vacuum

The surface properties of the xerogels were observed by BET N₂ physisorption. The list of BET surface areas, average pore size, and pore volumes of silica-based bulk xerogels is writen in Table 2. The highest BET surface area obatained from silica-pectin xerogels (2.5 to 4 time higher than organosilica and pure silica xerogels), respectively. The BET surface areas of pure silica and organosilica in this work are slightly lower than pure silica and organosilica xerogels prepared by Elma *et al.*, [50] and Elma *et al.*, [47], respectively. It is due to the pure silica xerogels synthesized by previous study was prepared under vacuum using conventional thermal processing (CTP) technique. In other hand, the organosilica by Elma *et al.*, [47] fabricated at different calcination temperature of 250 °C. Beside that, the pore volume of silica-pectin xerogels also shows higher than other samples of 0.71392 cm³/g (Table 2). These results caused by the addiction of pectin template into silica sols and brings the xerogel tend to form mesoporous. That is proved by the average pore size of silica-pectin also wider than pure silica and organosilica pore size about 3 fold times bigger. The bigger average pore size of silica-pectin will be collided with the membrane performance.

Table 2						
Surface properties of bulk silica-based xerogels in varied type sols						
Samples	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Pore size (nm)			
Pure Silica	101.1553	0.054508	2.1554			
Organosilica	221.9961	0.119156	2.147			
Silica-Pectin	470.2607	0.71392	6.07254			

From the results shown in Figure 5, it was apparent that TMP influenced the pure water permeability, and the performance of this membrane for treating peat water was investigated on Figure 6, using multiple TMPs (1-3 bar). The membrane permeability is an important factor to be considered during the ultrafiltration process. It is performed that the pure water permeate flux is increased by the raising of TMP. The gradient of the dash line represented pure water permeability of the silica-based membranes, which the results shown sufficient fit with Darcy's law, with R² of 0.976, 0.9737 and 0.9868, for varied multichannel membrane that coated by pure silica, organosilica and silica-pectin sols, respectively. These results indicated that transmembrane pressures ranging from 1 to 3 bar was very suitable for further ultrafiltration experiments.



Fig. 5. The effect of the transmembrane pressure (TMP) on the pure water permeability of various multichannel membranes thin film coating

3.3 Multichannel Silica-Based Membranes Performance for Peat Water Ultrafiltration

From the results shown in Figure 6, it was apparent that TMP influenced the permeate flux, and the performance of all multichannel silica-based membranes for treating peat water at different TMP. It is observed that the permeate fluxes gradually arise by increasing TMP. It is due to the driving force from TMP contributes to improve the hydraulic efficiency on ultrafiltration membrane and led the fluxes to increase [56]. The highest permeate flux was obtained from multichannel silica-pectin membrane at TMP 3 bar (96 Lm⁻².h⁻¹.bar⁻¹).

Furthermore, all UV₂₅₄ rejection of multichannel silica-based membranes obtained high as shown in Figure 6 at variation of TMP from 1 to 3 bar. The highest UV₂₅₄ rejection obtained for multichannel organosilica and pure silica membranes over silica-pectin membrane at all TMP minimum 88%. Meanwhile, the multichannel silica-pectin membrane performs the lower UV₂₅₄ rejection of 71% (Figure 6). It could be explained the average pore size of multichannel silica-pectin membrane was bigger than other membrane 3 times. Therefore, the rejection of UV₂₅₄ of multichannel silica-pectin membrane become lower than other membranes rejection.



Fig. 6. Membrane performance of multi-channel silicabased membranes in varied thin film coating at different transmembrane pressure (TMP) 0.1, 0.2, and 0.3 bar

Figure 7 demonstrated the normalized permeate fluxes (J_{α}) of multiple type silica-based membrane as function of filtration time at TMP 3 bar operation condition of peat water as feed. From all of normalized permeate fluxes multichannel silica-based membrane that consists of pure silica, organosilica and silica-pectin exhibit excellent stability for 180 min filtration time of peat water. As shown in Figure 7, the multichannel organosilica membrane performs fluctuated maybe due to the noise of the data, even though the trends of normalize permeate flux still ecellent condition for over 3 hours operation. It proves that all of multichannel silica-based membranes have excellent performance for treating peat water via ultrafiltration process.



Fig. 7. Performance of silica based membranes on normalized permeate flux variation over time, for various multi-channel thin film coating, at TMP 3 bar

Henceforth, Figure 7 shows excellent performance using all of multichannel silica-based membranes applied for peat water ultrafiltration for over 3 hours. This result indicates that there is no fouling occurred during time operation. Previous work was reported the multichannel ceramic membrane at TMP 1 bar for cattle wastewater ultrafiltration under feed temperature 30 °C shows sharply declining data on normalized permeate flux in the initial 10 min operation time [57]. If comparing to this result, the finding of silica-based membranes performance is much more stable and showing that there is no fouling found during ftreating of peat water for 3 hours of filtration process.

4. Conclusions

Silica based xerogels and multichannel membranes were prepared from the sol-gel method and inner coating technique. The silica based xerogels in this work consisted of pure silica, organosilica and silica-pectin thin film which coated onto multi-channel alumina substrate (four bores) that is calcined under vacuum condition. The application of various multi-channel silica based membranes coating were investigated for treating peatland water via ultrafiltration at multiple transmembrane pressure (TMP) from 1 to 3 bar. The functionalization of all silica-based xerogels were found that there are silanol, siloxane and silica-carbon. While deconvolution of peak area ratio of silanol over siloxane is obtained lower at silica-pectin xerogel. That means that the silica-pectin has lower hydrophilic silanol species than other materials. Also, it brings out the silica structures become more stable attaching in the water. The highest BET surface area, pore volumes and average pore size also obtained from silica-pectin xerogels. It is about 2-4 folds time than pure silica and organosilica. The performance all of multichannel silica-based membrane performs stable for operation test over 180 h at peat water ultrafiltration at TMP 3 bar. Overall, all off membranes shows high natural organic matter rejection by UV₂₅₄ parameter (above 70%) for high TMP. Due to this, the performance of multichannel silica-based membranes are very good for treating peat water treatment via ultrafiltration process at TMP from 1 to 3 bar.

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