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Single Vs Multichannel Silica-Pectin Ultrafiltration Membranes for Treatment of Natural Peat Water

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ABSTRACT

A comparative study was undertaken to investigate the potential of silica-pectin ultrafiltration membrane on varied operation pressure (0.1-0.3 MPa). Cross-flow ultrafiltration works were explored with tubular alumina substrates in single and multichannel configuration. The silica-pectin sol was prepared from TEOS as silica precursor by sol-gel method, afterward pectin was templated into the silica sol to induce carbon on it. Thereafter, both of single and multichannel alumina substrates was coated by the silica-pectin sol via inner coating technique. The study exhibited better performance of multichannel silica-pectin ultrafiltration membrane (with 4 bores) compared to the single channel membrane in terms of the permeate quality of UV_{254} rejection, as well as the permeate flux. The UV_{254} rejection of multichannel silica-pectin ultrafiltration membranes conducted over 84% in compliance with highest permeate fluxes of 234.3 $L.m^{-2}.h^{-1}.bar^{-1}$ at high operation pressure (TMP 0.3 MPa). The multichannel ultrafiltration membrane permeate flux in this work is slightly high 60-75% over single channel. Hence, both single and multichannel silica-pectin membranes exhibited steady neutralized permeate fluxes under TMP 0.3 MPa conditions for natural 100% peat water concentration, which indicates that fouling is not occurred during filtration time up to 60 min. Therefore, multichannel silica-pectin ultrafiltration membrane in this work is success to treatment of natural peat water, that the natural organic matter removal was found to be fit for uses as clean water sources.

1. Introduction

South Kalimantan-Indonesia is located in southern part of Borneo which has 4.3 million population in 2020. Almost half of the population live in remote rural areas, which agriculture is the main source of income. They are often geographically isolated and lack access to electricity and clean water supply. It is essential to ensure the availability of local source of water supply and even develops new potential sources of water such as from peatland to overcome future water shortages [1]. Tropical peatlands are most extensive in Southeast Asia where they are concentrated on the

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island of Sumatera and Borneo. Peat water is abundantly found in peatland due to availability of underground water and rainwater. Peat water is commonly used for domestic usage in rural coastal areas households which are yet to receive clean water from the municipal water supply. However, direct consumption of peat water is not advisable, hence, a simple and versatile water treatment system such as membrane separation is useful in this case [2].

Ultrafiltration is a promising technology for the pre-treatment or primer process of surface water in water purification plants [2-5]. Among the technologies, ultrafiltration membrane provides the interesting features of less energy consumption, simple operation, lower footprint, and lower cost [6-11]. Inorganic membrane such as silica is promising to use for ultrafiltration. Silica membrane offers many advantages such as good molecular sieving, resistant to high temperature and chemical, robustness, and more antifouling than polymer-based membrane. In addition, there is multiple type of tubular membrane support which commercial for separation application, i.e., single channel and multichannel. Literally, single channel alumina membrane support more familiar to utilize among researcher and industry, due to convenient to coating. However, single channel membrane tubular has lower surface area than multichannel. Due to that multichannel membrane might potentially to developed and implemented for scaled up to provide clean or potable water among rural communities.

Basically, silica membrane was prepared by simple sol-gel method and subsequently the silica sol was coated onto alumina membrane tubular support to fabricate. Based on material, carbon templated silica membranes are considerably used for water treatment due to enhanced the strength of the silica membrane [12-14]. Moreover, Carbon templated silica membranes prepare with the addition of citric acid can also be utilized to produce carbon groups to strengthen the matrix structure on thin-films as in the previous studies [15, 16]. The pure silica membrane for natural wetland water firstly introduced by Elma and Assyaifi [17]. It is fabricated and sintered via rapid thermal process and only need a day to produced silica thin film for four layers [18, 19]. Other study used pectin, citric acid and triblock copolymer P123 as a carbon source which was inserted into a silica matrix to increase hydrostability [1, 20-22]. Carbon from pectin enable to increase silica membrane performance applied for photocatalytic-pervaporation of wetland saline water have been published by Assyaifi*, et al.* [23]. Previous studies have been success to fabricated of silica-pectin membrane for application on wetland saline water via pervaporation method [8, 24-27].

Multichannel membranes offer more than one bore which can be increases the surface area of the membrane and resulting in higher flux than single channel membrane [28]. The properties of tubular multichannel has: (1) a pore size between 8-10 nm; (2) porosity ranging from 30-35%; and (3) mechanical strength between 5000-6000 MPa [28]. Preparation of PDMS (polydimethylsiloxane) layer coated the inner surface of a ceramic HF membrane has been successfully fabricated using a coating/cross flow method [29]. This method consisted of two type method i.e., static coating and cycled flow coating. The PDMS solution was adsorbed on the ceramic surface and the penetrated into the pores of the hollow fiber substrate to form transition layer in the interface at specific time for static coating. Meanwhile, after static coating, a gentle crossflow was introduced to remove the excess PDMS solution using syringe within the fiber bores. The presence of crossflow coating able to maintain coating layer thickness become thinner than absence of it. Other than that, calcination process also has important role for silica matrix tuning.

Elma*, et al.* [30] has investigated that vacuum calcination resulted the silica matrix become more mesoporous with higher carbon yield compared to N_2 calcination, that resulting in relatively superior performance during the desalination process. This study aims to investigate the potential of silica-pectin single and multichannel ultrafiltration membrane on varied operation pressure (0.1-0.3 MPa). Beside that also to demonstrate the membrane performance for natural peat water treatment which

prepared by inner coating with silica-pectin thin film only on the inner bore through the inner coating method.

2. Methodology

2.1 Chemicals and Materials

Peat water was collected from Jalan Suka Maju, Banjarbaru, South Kalimantan-Indonesia which pointed at GPS coordinate of -3.405418, 114.719799. The ultrafiltration feed water was prepared by diluted peat water with demineralize water into several concentration (25-100% of peat water). Meanwhile, the primer chemicals and reagents utilized consisted of tetraethyl orthosilicate (TEOS, 99%; Sigma-Aldrich) as a silica precursor, ethyl alcohol (ethanol, 96%) purchased from local distributor PT. Sumber Kita Indah, Bekasi-Indonesia and demineralized water were used as solvent to synthesize of silica-pectin sols. Nitric acid (0.00078 N HNO_3), and ammonia (0.0003 N NH_3) were obtained from Merck as acid and base catalyst, respectively. Pectin was extracted from local banana peel wastes with detail procedure on Isdayanti*, et al.* [31] works reported. Microporous alumina substrates single channel and multi-channel 4 bores (α-Al_2O_3 tubular support) were obtained from Ceramic Oxide Fabricators, Bendigo-Australia.

2.2 Silica-Pectin Sols Preparation and Characterization

Preparation of silica-pectin sols were conducted via sol gel method with the detail procedure can be found in our previous work reported by Pratiwi*, et al.* [32]. The final molar ratios of silica-pectin sols of TEOS: ethanol: HNO_3:H_2O:NH_3:pectin were obtained become 1:38:0.0008:5:0.0003:0.00065. Afterwards, the silica-pectin sols dried at 60 °C for 24 h. The dried gel of silica-pectin then grounded into powder which named as xerogels. The silica-pectin xerogels calcined at 300 °C using vacuum furnace for an hour. The silica-pectin xerogels were characterized by FTIR (Fourier Transform Infrared) – Bruker Alpha spectrometer at wavenumber range of 600–1400 cm^{-1} to functionalize its xerogels properties and TGA (Thermogravimetric Analysis) to investigate the carbonization.

2.3 Fabrication and Characterization of Silica-Pectin Membranes

Silica-pectin membranes were prepared by inner coating method onto varied membrane support i.e., single and multichannel alumina substrates. The membranes were contained 4 coating layers, which every layer was calcined under vacuum condition at 300 °C. Illustration of inner coating set-up of membrane fabrication can be seen in Figure 1 (a). The morphology of silica-pectin membranes was also investigated by scanning electron microscopy (SEM, ZEISS).

2.4 Performance of Silica-Pectin Membranes Ultrafiltration

Figure 1 (b) illustrates the laboratory-scale ultrafiltration set-up equipped with a tubular module to place silica-pectin membrane for multiple configuration membrane (single and multichannel). Ultrafiltration (single and multichannel) silica-pectin membranes were operated by cross flow system. The filtrations were conducted at variable TMP (transmembrane pressure) 0.1-0.3 MPa for 60 min filtration time at room temperature (25 °C ± 2 °C). A booster pump generated the pressure, and the permeate volume was collected every 5 min.

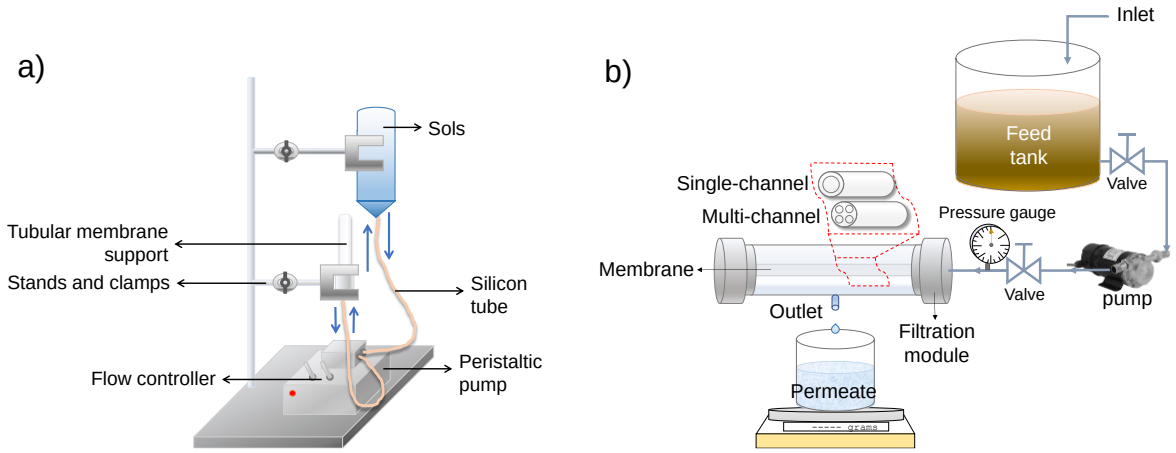

Fig. 1. Schematic of set up for (a) membrane inner coating; and (b) peat water ultrafiltration experiment

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

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

$$R=(C\_f-C\_p)/C\_f \times 100\%, \quad (2)$$

3. Results and Discussion

3.1 Characteristic and Morphology of Silica-Pectin Membrane

Functional groups of silica-pectin xerogel calcined at 300 °C under vacuum condition shows in Figure 2 to represent the FTIR spectra in range of 600-1400 cm^{-1}. The pectin from banana peels templated into silica sols resulted to form Si-C stretching vibration at wavenumber 802 cm^{-1}. The deconvoluted peaks of Si-C bands are arise due to the wagging mode of carbon from pectin attached to Si which similar to previous work have been reported [33]. Besides that, the dual acid-based catalyst also dictated the structure of the formed silica network. Si-OH (silanol) and Si-O-Si (siloxane) groups were formed via hydrolysis and condensation reactions during the sol-gel process. The Si-O-Si stretching linear vibration also can be encountered at 1200 and 1067 cm^{-1} bands as reported in previous works on silica membranes-based materials [34-36]. Si-OH bands appear at shoulder of siloxane peak near 967 cm^{-1}.

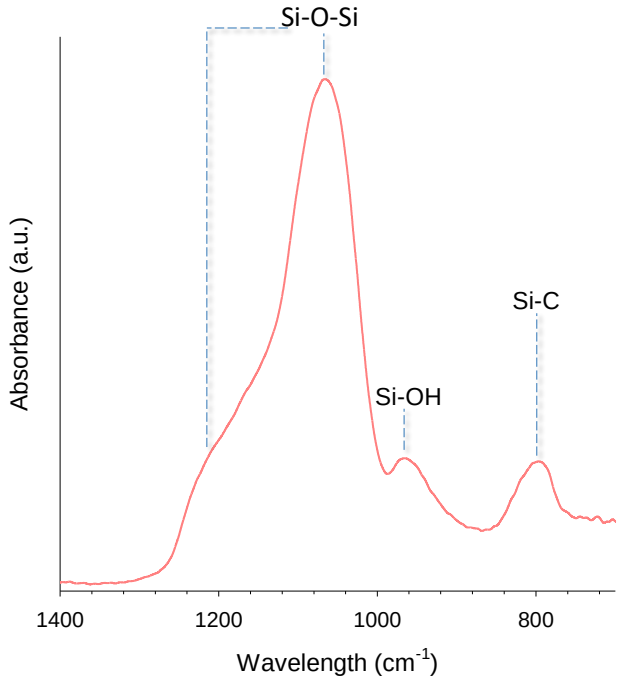

Fig. 2. The FTIR spectra of silica-pectin xerogel

Siloxane is tailored by solvent condensation reaction via sol-gel method. Meanwhile, the silanol was obtained from the hydrolysis reaction at first-step process. Addition of acid catalyst to the silica sols promoted the rate of the hydrolysis reaction. The addiction of pectin into silica matrices contributes to enhance the hydrostability of silica matrices proved by the presence of Si-C bond (Figure 2). It is appropriated to the latest study reported elsewhere [34]. This finding can be seen as good indication to improve silica network and perform more stable of membranes for water desalination.

Furthermore, TGA analysis was conducted to examine of carbonaceous matrix of silica-pectin xerogel of Silica-P123. The silica-pectin xerogel without calcination was pre-calcined from room temperature to 800 °C. Figure 3 exhibits the mass lost profile of the silica-pectin xerogel as function of temperature series. The result performs the overlapped decomposition steps occurred in three stages. The first stage taken place at temperature <150 °C with mass losses of 15 wt%. This stage performs the evaporation phase of solvent such as ethanol and demineralize water. The second and third stages examined at temperature between 100 to 530 °C (8 wt.%) and up to 530 °C (4 wt.%), respectively as displays on Figure 3. In this work, the volatile compound quite decomposed during first and second stages due to at temperature up to 100 °C, the water undergo to desorption in porous structure and indicating as weight loss start point [37, 38]. The final stage predominantly related with the main decomposition of chemical and recombination within the pectin as it carbonizes.

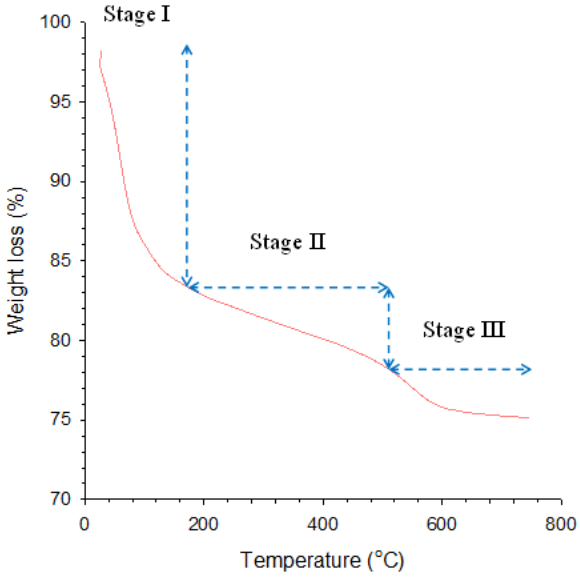

Fig. 3. Thermal gravimetric mass loss curves of silica-pectin membrane

Figure 4 shows SEM photograph of surface area indicates the morphology structure of silica-pectin membrane in different configuration of (a) single channel and (b) multichannel (4 bores). Single channel silica-pectin membrane exhibits non ideal homogeneous coating compare multi-channel membrane surface. It is due to the inflow of the silica-pectin sols into the inner surface of tubular substrate through a single channel [39]. The view photograph of the membranes is presented in high resolution to display the effect of the coating on inner surface of the membrane. In both of Figure 4 (a) the coated membrane of single channel and Figure 4 (b) multichannel configuration can be seen the top layer have the roughness and boulders of the alumina particles substrate protruding on silica-pectin membrane surface. Moreover, the top layer silica-pectin membranes displays not delicate caused by its membranes were coated direct onto microporous substrate without interlayer similar to work report by Elma*, et al.* [40].

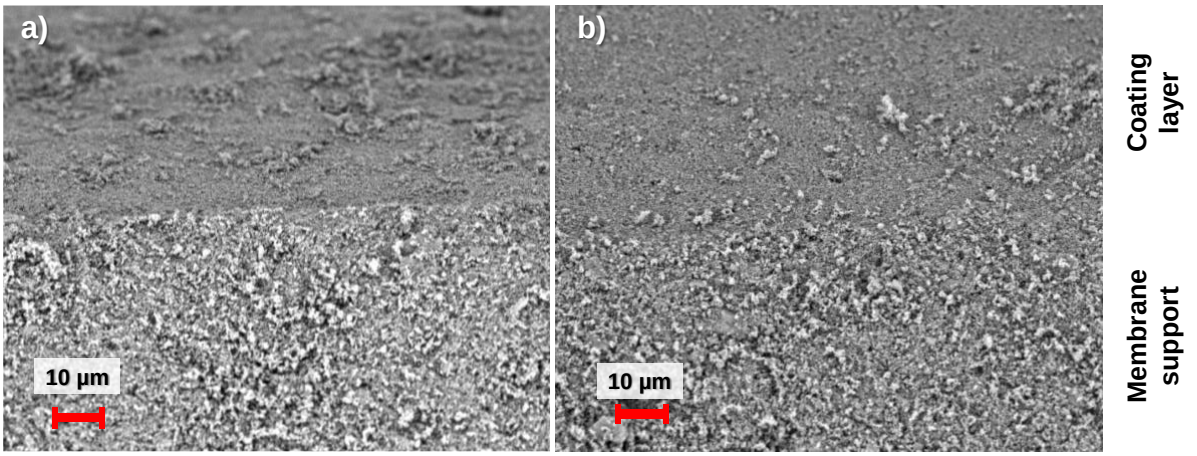

Fig. 4. SEM photograph of silica-pectin membrane for multiple configurations of (a) single channel and (b) multichannel (4 bores) by inner coating method

As shown in Figure 4, both of single and multichannel surface layer exhibited there are the difference morphological structure between coating layer and membrane support. The silica-pectin

coating layer observes tighter compared the single and multichannel membrane support. It is due to the silica-pectin sols was filled in the channel or bore side of the alumina substrates. The sols were adsorbed on the membrane support surface and then penetrated onto the pores of the substrate to form a transition layer in the interface. In contrast, SEM photograph of multichannel silica-pectin membrane can be seen denser as shown in Figure 4 (b). There was a clear transition layer between the silica-pectin coating layers and the alumina substrate. This could be a reason for the lower velocities in the porous support structure in the region between outer bore channels [41]. Therefore, silica-pectin sols facile to infiltrate into multichannel substrate. This transition layer provided the membrane with sufficient interfacial adhesion, resulting in a silica-pectin layer that tightly covered the inner surface of the alumina substrates single and multichannel configuration with no delamination [29].

Furthermore, the morphology structure and physical properties of silica-pectin membrane with difference configuration (single and multichannel) regarding to affect the membrane performance. Table 1 summarizes the physical properties of silica-pectin membrane in single and multichannel membrane configuration. The multichannel silica-pectin membrane has higher filtration active area about 25% over single channel membrane. As theory, the highest filtration active area leads the performance of membrane could be raised.

Table 1
Physical properties of silica-pectin membrane in multiple single and multichannel configuration

Properties	Single channel	Multichannel (4 bores)
Outer diameter (mm)	8	8
Channel diameter (mm)	6	2
Length of tube (mm)	35	35
Average substrate pore size (µm)	0.1	0.1
Average silica-pectin layer pore size (nm)	6.1	6.1
BET surface area of silica-pectin layer (m^2g^{-1})	470	470
Pore volume of silica-pectin layer (cm^3g^{-1})	0.71	0.71
Channel thickness (mm)	1	1
Filtration active area (m^2)	6.59	8.79

From the results in Figure 5, it was apparent that TMP influenced the demineralized water permeability, and so the performance of this membrane for treating peat water was investigated, using different TMPs (0.1, 0.2 and 0.3 MPa) and membrane configuration (single and multichannel membrane). The multiple membranes were chosen for the ultrafiltration process, as membrane permeability is an important factor to be considered during the UF process. It displayed the demineralized water permeate flux increased with increasing TMP. The gradient of the dash line represented demineralized water permeability of the silica-pectin membranes, and as inferred, the results shown sufficient fit with Darcy's law, with R^2 of 0.962 and 0.9757, for single and multichannel membrane coating, respectively. These results indicated that TMP ranging from 0.1 to 0.4 MPa was suitable for further UF experiments.

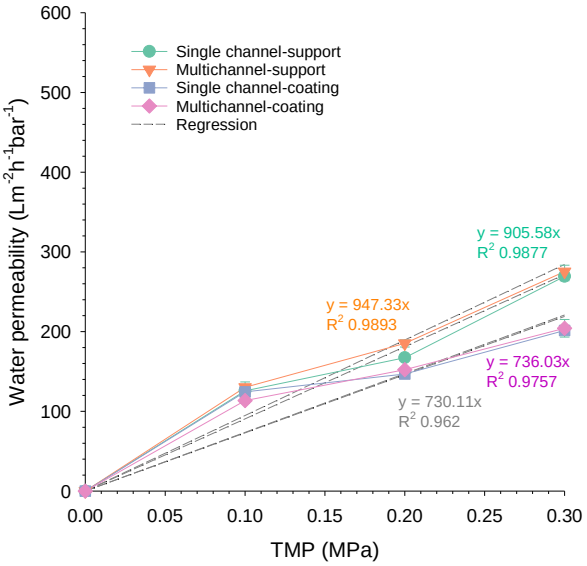

Fig. 5. The effect of the transmembrane pressure on the water permeability of single and multichannel silica-pectin membranes

3.1 Performance of Silica-Pectin Membrane for Peat Water Ultrafiltration

Figure 6 shows the effect of peat water concentration (25, 50, 75 and 100 % peat water) on the permeate fluxes with respect to membrane configuration (single and multichannel) and varied TMP.

From the results in Figure 6, it was apparent that peat water concentration influenced the permeate flux, and so the performance of both single and multichannel membrane for treating natural wetland water at different TMP. It is observed that the permeate fluxes gradually decline by increasing peat water concentration. This behavior was thought to be since natural organic matter (NOM) as foulants in the high concentration feed caused performance of membrane become decreased. The presence number of NOM in peat water led to blocking membrane pore namely fouling. Commonly, the molecular size of NOM is 1-100 kDa which greater than silica-pectin membrane pore size about ~6 nm [42, 43]. Due to that deposition and restrained on top of membrane layer caused water flux gradual drop.

Multichannel silica-pectin membrane was apparent highest permeate flux over single channel for all peat water concentration (25-100%) at multiple TMP as seen on Figure 6 (a), (b), and (c). The permeate fluxes of multichannel silica-pectin membrane is average higher of >60% than single channel for all TMP 0.1, 0.2 and 0.3 MPa. In addition, it is examined that the permeate fluxes increasing as TMP increased from 0.1 to 0.3 MPa. The highest permeate flux of multichannel silica-pectin membrane received of 234.3 $L.m^{-2}.h^{-1}.bar^{-1}$ at TMP 0.3 MPa for treating 25% peat water concentration. The lowest concentration of organic substances in feed water contributed to improve hydraulic efficiency on ultrafiltration membrane [44].

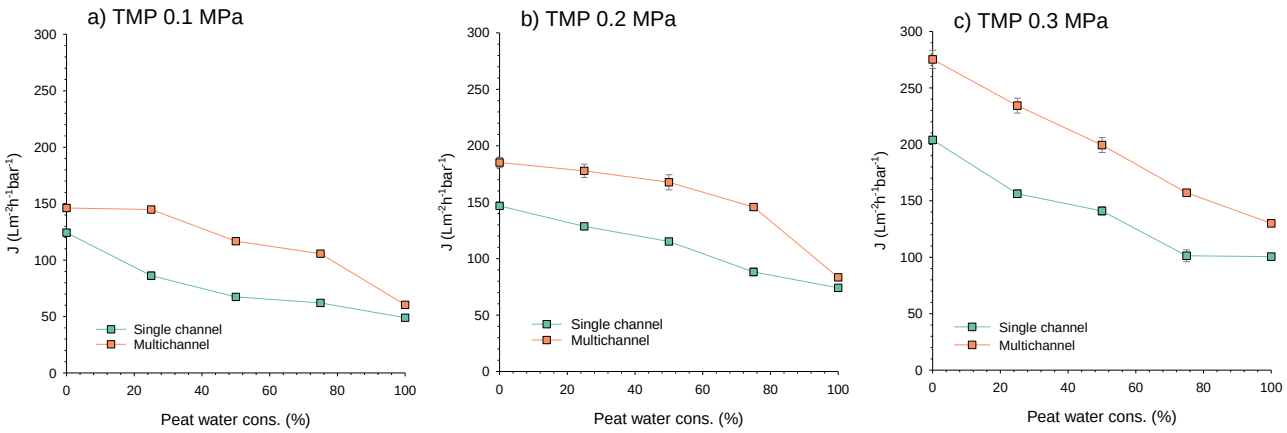

Fig. 6. The effect of peat water concentration on the permeate fluxes of single and multichannel silica-pectin mem-branes at different TMP (a) 0.1; (2) 0.2; and (3) 0.3 MPa

The normalize permeate fluxes (J_α) against time of the multiple membranes as function of filtration time shown in Figure 7 at TMP 0.3 MPa operation condition for 100% concentration of peat water. Both of single and multichannel silica-pectin membrane normalized permeate fluxes perform excellent stable for 60 min operation time of peat water ultrafiltration. As displayed in Figure 7, the multichannel silica-pectin membrane exhibited the slightly lower in its normalized permeate flux over the single channel membrane, in spite of extremely steady. Notwithstanding a few declines of the normalized permeate flux on multichannel supposed caused by the silica-pectin layer of multichannel is slightly tighter than single channel. It proved as resulted of SEM image on Figure 4 (b), despite of multichannel silica-pectin membrane has larger active surface filtration area.

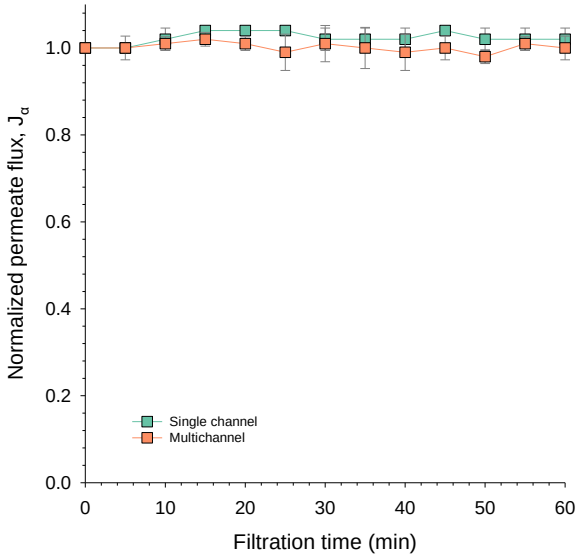

Fig. 8. Silica-pectin membranes normalized permeate flux variation over time, for various single and multichannel, TMP 0.3 MPa

Interestingly, the results of the all configuration silica-pectin membrane either single and channel on normalized permeate flux exhibit do not decline overall an hour filtration time. It denotes the all silica-pectin membrane configuration still has good performance. That means both single and multichannel membrane there were not indicated to occur fouling overall. Previous study reported the multichannel ceramic ultrafiltration membrane at TMP 0.1 MPa under feed temperature 30 °C of

cattle wastewater performed sharply declined of normalized permeate flux in the initial 10 min operation time [45]. This work finding is more potentially for treating peat water with no fouling at 60 min operation time.

Hence all UV_{254} rejection of single and multichannel silica-pectin membranes obtained relatively high as shown in Figure 8 at variation of peat water concentration (25, 50, 75 and 100%) at overall TMP (0.1, 0.2 and 0.3 MPa). The highest UV_{254} rejection obtained for single channel membrane over multichannel silica-pectin membrane at all TMP and variation of peat water concentration minimum 81%. Meanwhile, the multichannel silica-pectin membrane performs the lower UV_{254} rejection of 72% (Figure 9). It could be explained by the single channel has a lower mass transfer resistance compared to the multichannel due to the smaller path length towards the permeate [46]. The filtration rate of the single channel might be slightly larger than that of the multichannel that would result in a faster growing concentration polarization layer. Therefore, single channel membrane has higher rejection compare with multichannel silica-pectin membrane.

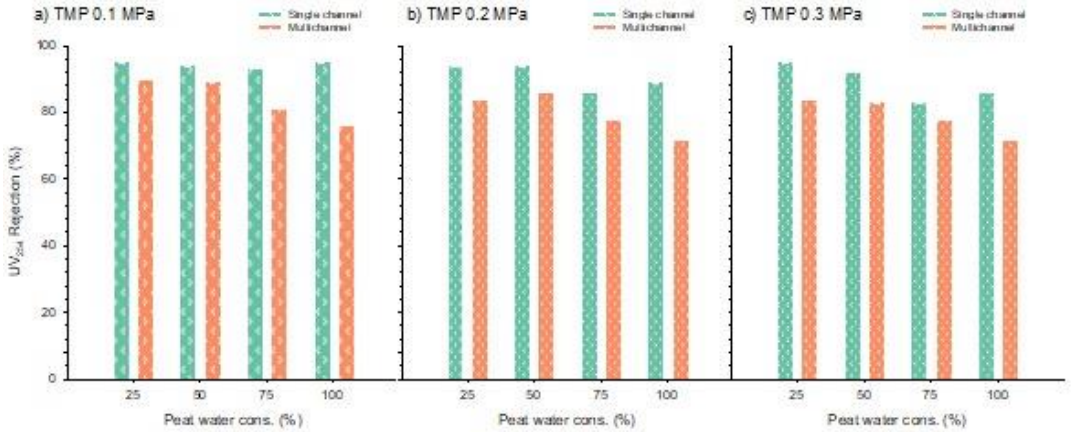

Fig. 9. Silica-pectin membranes UV_{254} rejection variation over peat water concentration, for various single and multi-channel at different TMP of (a) 0.1; (b) 0.2; and (c) 0.3 MPa

4. Conclusions

The application of single and multichannel silica-pectin ultrafiltration membranes to peat water treatment were tested and the results exhibited that, firstly, the excellent performance obtained from single channel membrane at TMP 0.3 MPa for 100% peat water concentration (natural peat water). Secondly, the membranes exhibited stable operation under the excellent conditions above, which indicates that fouling is not occurred during filtration time up to 60 min. Finally, the rejection of UV_{254} as representative of natural organic matter parameters were quite high of 95% by single channel membrane at lower TMP 0.1 MPa and 86% at 0.3 MPa. It is concluded that single channel silica-pectin membrane shows a better result than multichannel silica-pectin membrane according to the fluxes and rejection performances for natural peat water treatment.

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