Vol. 26 No.1 (Januari 2025) 090-096
e-ISSN 2548-6101 p-ISSN 1411-318X

Jurnal Teknologi Lingkungan

Journal Homepage: ejournal.brin.go.id/JTL

Lab Scale Investigation of Inline Powdered Active Carbon-Ultrafiltration Membrane as Pretreatment for Seawater Reverse Osmosis

Investigasi Skala Laboratorium Karbon Aktif-Ultrafiltrasi Membran sebagai Prapengolahan unit Reverse Osmosis Air Laut

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ARTICLE INFO

Article history:
Received 9 February 2024
Accepted 9 August 2024
Published 31 January 2025

Keywords:
Desalination
Powdered Activated Carbon (PAC)
Hybrid Membrane Process
Membrane Performance
Membrane Fouling

ABSTRACT

Reverse osmosis is a technology to that treat treats seawater as an alternative source for of drinking water supply. Several studies confirmed that pretreatment stages, designed to tackle high salinity and organic material in seawater, contribute significantly to the reliability of SWRO technology. Despite its effectiveness, declination of the performance of SWRO after several operation times resulted in potential improvement. Enhancement of pretreatment process to increase the performance of SWRO is necessary to be investigated. A combination of powdered activated carbon and ultrafiltration (PAC/UF) is employed at a lab scale experiment using real seawater matrix collected from an outlet dissolved air flotation unit as SWRO pretreatment stage. The Experiment was conducted employing a polyethersulfone (PES) membrane with an average pore size of 30 nm at dead-end operation with constant flux of 60 and 120 $L/m^2 \cdot h$*, respectively. For activated carbon, commercial coconut shell-based PAC was added inside the membrane holder and directly deposited on top of the membrane surface. The experiment was conducted by comparing a single filtration cycle operation using UF with/without activated carbon. UF membrane filtration performance, as well as membrane retention, was observed. PAC/UF resulted in higher removal of organic concentration (UV-Vis) (93-96%) compared to UF (14-43%). Both experiments resulted in high removal up to >90% for turbidity and chemical oxygen demand. Nevertheless, insignificant removal of salts concentration (<5%) was observed. In terms of performance, combined PAC/UF revealed better performance despite a thicker layer in comparison to UF membrane at a similar filtered specific volume of water production. Therefore, a combination PAC/UF at constant flux operation was found to be promising and reliable pretreatment for SWRO.*

INFORMASI ARTIKEL

Histori artikel:
Diterima 9 Februari 2024
Disetujui 9 Agustus 2024
Diterbitkan 31 Januari 2025

Kata kunci:
Desalinasi,
Powdered Activated Carbon (PAC),
Proses Hibrid,
Performa Membran,
Fouling Membrane

ABSTRAK

Kebutuhan air minum mengalami peningkatan seiring bertambahnya penduduk, tetapi sumber air bersih mengalami penurunan. Hal ini berdampak terhadap pemanfaatan sumber alternatif air laut untuk pemenuhan kebutuhan air minum dengan teknologi reverse osmosis. Walaupun dapat diandalkan untuk mengurangi salinitas tinggi dan kandungan organik air laut, unit pengolahan seawater reverse osmosis (SWRO) mengalami penurunan kinerja setelah pengoperasian dalam kurun waktu tertentu. Salah satu upaya adalah dengan peningkatan proses prapengolahan untuk SWRO. Pada penelitian ini, kombinasi Karbon Aktif Serbuk dan Ultrafiltrasi mempergunakan air laut yang berasal dari outlet unit prapengolahan pada instalasi SWRO. Percobaan filtrasi dilakukan pada percobaan skala laboratorium mempergunakan membran polyethersulfone (PES) dengan ukuran pori 30 nm pada proses dead-end dengan fluks konstan 60 dan 120 $L/m^2 \cdot jam$. Karbon aktif bubuk komersial berbahan dasar tempurung kelapa diletakan di atas permukaan membran. Percobaan dilakukan dengan membandingkan operasi siklus filtrasi tunggal menggunakan UF dengan dan tanpa karbon aktif. Parameter pemantauan kualitas air seperti kandungan organik yang dipergunakan antara lain UV-Vis, kekeruhan, kandungan oksigen kimiawi (COD), dan koduktivitas. Penelitian ini menjelaskan bahwa retensi membran PAC/UF menghasilkan penyisihan konsentrasi organik (UV-Vis) yang lebih tinggi (93-96%) dibandingkan dengan hanya mempergunakan UF (14-43%). Untuk kekeruhan dan kebutuhan oksigen kimiawi, kedua percobaan menghasilkan penyisihan yang tinggi hingga >90%. Jika mengacu pada performa filtrasi membran, volume produksi air yang dihasilkan sebanding untuk percobaan hanya dengan UF dan PAC/UF meskipun lapisan PAC/UF lebih tebal. Berdasarkan temuan pada penelitian ini, dapat disimpulkan bahwa kombinasi PAC/UF pada operasi fluks konstan dapat dipergunakan prapengolahan yang dapat diandalkan untuk SWRO.

1. INTRODUCTION

1.1 Background

Indonesia's area of 7.81 million km^2 consists of 64.97% sea and 35.03% land (Yasir, 2019). The large sea level in Indonesia can be an alternative solution as a source for potable water to meet higher potable water demand, especially in areas where surface water is heavily polluted and tend to be close to the sea, as well as in areas with limited land (Kunzmann, 2018). However, high salinity and high organic carbon concentration seawater characteristics are the major obstacles to drinking water production (Kim, 2013; Kennedy, 2021). The technology for seawater desalination points out the selection of Seawater Reverse Osmosis (SWRO) (Maxime, 2023). Despite being reliable, after several years of operation, the performance of (SWRO) tends to decline and necessarily needs to be improved. Membrane fouling due to a combination of organic and high salinity was found to be the major problem that resulted in permeate flux decline, biodegradation of membrane materials that shortened membrane life, and system failure that increased operational cost due to the cleaning process as well as new membrane replacement (She, 2016). Related to membrane fouling, pretreatment is an important process in seawater desalination to reduce SWRO membrane loads. Pretreatment process can employ conventional technology or porous membranes such as microfiltration, ultrafiltration, and nanofiltration (Schippers, 2018). However, ultrafiltration membranes are a type of membrane that has a high level of contaminant separation and permeation flux and is effective in removing viruses compared to microfiltration to prevent biofouling (Ferrer, 2015; Gentile, 2018). Compared to nanofiltration membranes, ultrafiltration membranes have a higher flux and require lower operating pressure meaning more cost effective due to lowering operating cost and energi requirement (Selma, 2020). Another advantage of UF membranes in pretreatment is that they require less footprint than conventional methods (Li, 2018)

Among various kinds of UF membrane materials, PES UF is one of the polymeric materials membranes that are commercially available that commonly used for many science applications and water treatment due to their wide temperature & pH, hydrophilicity, and chemical resistance (Xiang, 2013). Previous studies reported that UF membrane was highly reliable for removing suspended and colloidal matter, even pathogenic bacteria and viruses, while limited removal efficiency for organic matter due to the pore size and the size of organic substance (Yang et al., 2022); Salinas-Rodríguez, 2021; Schwaller, 2021). Alternative pretreatment can be carried out using activated carbon (adsorption principle) by adsorbing a solid substance on the surface due to the attractive force of atoms or molecules on the surface of the solid substance (Takwanto et al., 2018). Adsorption using Powdered Activated Carbon (PAC) can be considered as an alternative pretreatment due to its low cost and high efficiency (Campinas et al., 2021). Previous study revealed that the addition of PAC as a UF membrane pretreatment was able to improve membrane performance regardless of the type of feed water (river or peat water, or wastewater) especially in removing organic matters such as Natural Organic Matter (NOM), Total Organic Carbon (TOC), UV-Vis (Schwaller, 2021; Elma, 2022). Furthermore, the addition of PAC before filtration does not cause irreversible fouling and reduces the impact of organic foulants (Darracq, 2015). In General, numerous study were conducted employing powdered activated carbon and ultrafiltration to treated water. However, lack of investigation employing combined PAC and ultrafiltration membrane as pretreatment of desalination SWRO.

1.2 Objective

The aim of this study is to analyze the removal efficiency and membrane performances of 30 nm Polyethersulfone (PES) flat sheet UF membranes compared to the precoating UF membrane with a inline Powdered Activated Carbon (PAC/UF) in dead-end circuits for clean/drinking water treatment in constant flux mode within the scope on a laboratory scale (lab scale) in order to investigate the combination as a pretreatment to reduce RO membrane fouling.

2. MATERIAL AND METHOD

2.1 Material & Characteristics

The feed water was collected between January and March 2023 from outlet dissolved air flotation (DAF) SWRO unit in North Jakarta, Indonesia, at average weather conditions. Therefore, no dilution factor needed to be considered. Characterization of feed water refers to drinking water standard parameters, including pH using a pH meter (HI 98107, Hanna, USA), turbidity by turbidimeter (TU-2016, Lutron, Taiwan), UV-Vis by spectrophotometer (DR-6000, Hach, USA) (wavelength 200-400 nm), and Chemical Oxidation Demands (COD) by spectrophotometer (DR-2000, Hach, USA). The Total Dissolved Solid (TDS), conductivity, and DO were measured with a multi-parameter meter (HQ40d, Hach, USA). The feed water had a neutral pH ranging from 6.5-7.3 and the dissolved oxygen (DO) value ranged from 6.57-6.89. The high concentration of dissolved solids salinity level was characterized by high TDS that ranged from 17,120-22,900 mg/L and conductivity that ranged from 25,500-32,800 μs/cm, meanwhile the turbidity relatively low with values 2.14-2.62 NTU. The organic compounds characterized by UV-Vis ranged from 0.45-1.07 ABS and COD with an average value of 140 mg/L. The low values in some parameters are due to seawater pretreatment by DAF that reduces the pollutant load.

Powdered Activated Carbon (PAC) was employed using a commercial product based on coconut shells. Prior to the experiment, the surface area and particle size distribution of the commercial PAC were characterized. The PAC has an average BET surface area of 835.237 m^2/g with a particle size distribution in range between 0.5 μm-6 μm. Furthermore, PAC was treated by washing with deionized water in order to remove undesirable contaminants. The PAC concentration used in this experiment is 0.5 mg based on capacity of the membrane holder.

2.2 Ultrafiltration and Hybrid Activated Carbon Experiments

The lab scale filtration setup (see Figure 1) was performed by filtration of feed water. During filtration, the feed tank was stirred to maintain the homogenous solution. The feed water was pumped into the Millipore membrane holder using a peristaltic pump BT300-2J by (LongerPump, China) at 1 and 2 RPM (Rotary per Minute) that represented equal to approximate of operation constant flux at 60 and 120 L/m²·h, respectively. Ahead of the membrane holder, the pressure gauge was installed to read the pressure difference during the filtration process. The change of the pressure occurred due to constant flux operation and collection. The filtration was conducted at dead-end mode operation and performed until the membrane become saturated and none of water permeate comes out from the filtration system (one cycle). The filtrated (permeate) water was measured using analytic scales and then recorded. The room temperature during experiments was also measured (26°C ± 2).

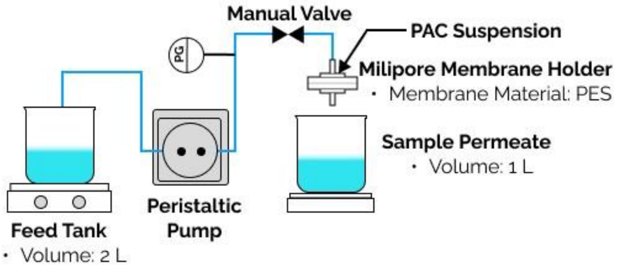

Figure 1. PAC-ultrafiltration dead-end experimental setup

For the membrane experiment, a flat sheet membrane was supplied by Sterlitech Corporation (United States) with a total membrane active surface area of 47 mm² and an average pore size 0,03 µm or 30 nm (see. Figure 2A). According to its manufacture information, the UF membrane is hydrophilic and has asymmetric pores characteristics. Prior to testing,a flat sheet membrane was soaked in 200 ppm NaOCL solution at pH 12 for 1 day to remove the contaminants/particles that block the membrane pores. The soaked membrane was rinsed with distilled water. The cleaned membrane was further installed inside the Millipore membrane holder. Afterward, the pure water was filtered through the installed membrane for 5 minutes with the purpose cleaning the system and ensuring the system was stable. After the system and the membrane were ready, the UF membrane started filtering feed water (DAF outlet) with the two different fluxes. During the experiment, the water permeate mass was recorded every 5 minutes by observing a digital scale and digital pressure gauge. The recorded mass and pressure were used to calculate flux and transmembrane pressure (TMP). In the case of hybrid PAC/UF, the experiment sequence of the filtration process was similar to the ultrafiltration mentioned before. The main difference is that an additional PAC of 0,5 mg was added on the top of the membrane as a precoat in the membrane holder system to run in inline UF/PAC filtration mode (see. Figure 2B). During the filtration, permeate water was collected and characterized to determine membrane retention.

Figure 2 PES ultrafiltration (A) and PAC-ultrafiltration membrane (B)

2.3 Membrane Performance Analysis Method

The collected data from the experiment were further calculated. In this study, several indicators were employed to analyze membrane retention and filtration performance. The membrane retention explained the removal efficiency of the parameters previously mentioned by comparing the initial concentration before filtration with the final concentration after filtration using equation (1) (Laksono, 2021). Samples were collected from outlet DAF water and permeates after one filtration cycle.

$$R = \left(1 - \frac{C_p}{C_f}\right) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where:

R = membrane retention
C_p = solute concentration in permeate,
C_f = solute concentration in feed solution

Furthermore, related to the membrane performance, the measured water flux and transmembrane pressure were employed to calculate water permeability using equation (2) and 3) and filtered specific volume see in equation (4) (Laksono, 2021).

$$W = \frac{J}{\Delta P} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

Where:

W : permeability,
J = water flux,
ΔP = transmembrane pressure,

The membrane permeability during filtration was normalized with respected to the membrane permeability at clean conditions (3) at certain filtered specific volume see in equation (4) (Laksono, 2021).

$$W' = \frac{W_{Vsp}}{W_0} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

$$V_{sp} = \frac{V_P}{Am} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

Where:

C_p = solute concentration in permeate,
C_f = solute concentration in feed solution
W_{Vsp} membrane permeability at certain filtered specific volume,
W_0 : permeability of clean membrane.

V_p : permeate volume, and

Am : surface area membrane.

Lastly, the calculated and measured data explained by equation (3) and (4) were further plotted as a curve (W vs Vsp) to understand the performance.

3. RESULT AND DISCUSSION

3.1 Performance of Ultrafiltration Membrane

The membrane performances for both filtration experiments (UF and PAC/UF) were plotted from the normalized permeability against specific filtered volume (see Figure 3 and 4) that was obtained during the experiments. The experiment was conducted two times using different feed concentrations to explain the reproducibility of the results. However, the plotted graph was chosen as the best curve performance for both UF and PAC/UF experiments. Related to the data of the graph, one might consider that the graphics fluctuated due to the unstable reading at digital pressure gauge and resulted the unstable permeability result. However, a certain pattern of decline in membrane permeability was revealed. A simple modeling approach was determined to emphasize the pattern of the membrane permeability decay curve.

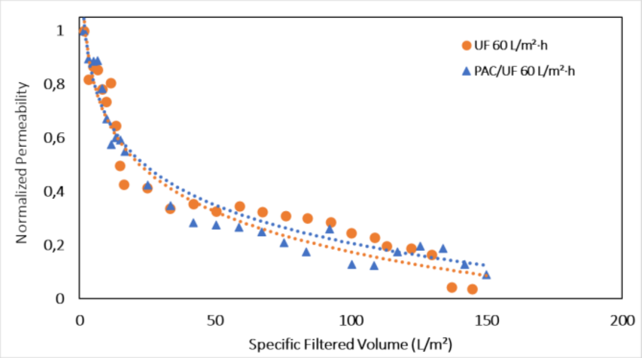

Figure 3. Membrane performance of UF and hybrid PAC/UF at 60 L/m²·h

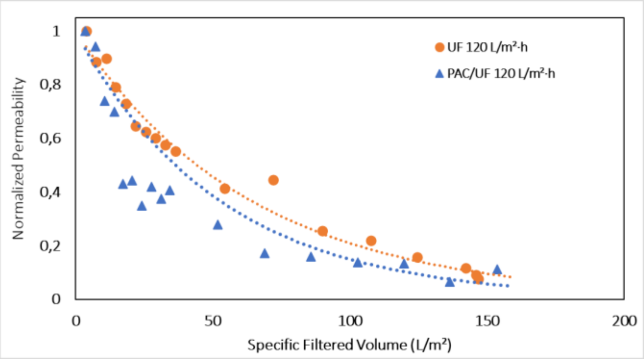

Figure 4. Membrane performance of UF and Hybrid PAC/UF at 120 L/m²·h

Generally, one might say filtration of ultrafiltration membranes at different flux had comparable filtration performance with the end of permeability in the range of 0,1 - 0,15 at a filtered specific volume of 150 L/m². In parallel, similar result was observed in case of PAC/UF membrane permeability decay (see. Figure 3 & 4). Despite comparable end permeability, the membrane decline curve for 60 and 120 L/m²·h revealed different fouling behavior. In the case of 60 L/m²·h, a steep and fast membrane permeability decay was found until the permeability decay reached 0,3 at the specific filtrated volume of 35 L/m², followed by a linear decrease of membrane permeability until the end of the filtration cycle. Based on the hydrodynamics of constant flux operation, phases of permeability decrease occurred as a representation of the fouling rate due to mass transfer and membrane selectivity (He, 2016). Steep and faster fouling in earlier phase represent the likelihood of standard and internal pore blocking phenomena. At same time, the later moderate permeability decline was attributed to intermediate pore blocking followed by formation multilayer and resulted in cake layer or cake filtration (Laksono, 2021). In parallel, a steep membrane permeability decrease reaches 0,3 with filtered specific volume of 75 L/m² was observed in early filtration phase in case of 120 L/m²·h followed by moderate permeability decline at later phase. By comparing two different fluxes, despite similar end permeability, a severe membrane fouling at the early phase of 60 L/m²·h than 120 L/m²·h was observed.

Further observation was conducted for PAC/UF at different fluxes. In case of 60 L/m²·h, steep permeability decline until reached 0,3 at specific filtered volume of 35 L/m² followed by linear moderate decrease at later phase. Furthermore, in the case of 120 L/m²·h, a steep permeability decline was observed until 0,3 with the specific filtered volume of 60 L/m² with a linear decrease at the later filtration phase. In comparison with UF Filtration, comparable permeability decline was observed in the early filtration phase during filtration of 60 L/m²·h. In the later phase, PAC/UF revealed slightly better with the end of permeability of 0,15 and UF with 0,1, respectively. On the other hand, in the case of 120 L/m²·h, stronger membrane fouling was observed during filtration of PAC/UF than Ultrafiltration without PAC. Despite better, considering several factors, one might consider that the results were comparable.

Interesting results were observed in the case of the combination process of PAC/UF. In principle, Membrane permeability is attributed to the thickness of the membrane. Additional activated carbon on top of the membrane (see Figure 2B) resulted in a thicker membrane layer (higher membrane resistance) and decreased water permeability (less filtered volume). However, the filtration results of PAC-UF revealed a comparable result with the normal ultrafiltration process. This finding indicates that the filterability of water was not affected by the secondary layer due to the deposition of PAC on top of the membrane.

3.2 Membrane Retention

Membrane performance was analyzed not only from filtration performance but also from membrane retention(see Figure 5). Membrane retention for both experiments (UF and PAC/UF) conducted high removal efficiency on turbidity and COD parameter with value (>90%) and increasing value of Dissolved Oxygen (DO) up to 7.9-8.2 mg/L that indicates better water quality due to decrease of pollutant (Franklin, 2014). High turbidity and COD retention were caused by the rejection of suspended solids by UF PES 30 nm mainly through physical interception (Wang, 2022). Additionally, the PAC, in combination with UF, had slightly higher removal efficiency on turbidity. The slightly higher removal efficiency might be explained by the fact that the modified tight UF PES membrane had removal percent of turbidity and COD with 99,6% and 99,8%, respectively, for wastewater treatment (Mansor, 2021).

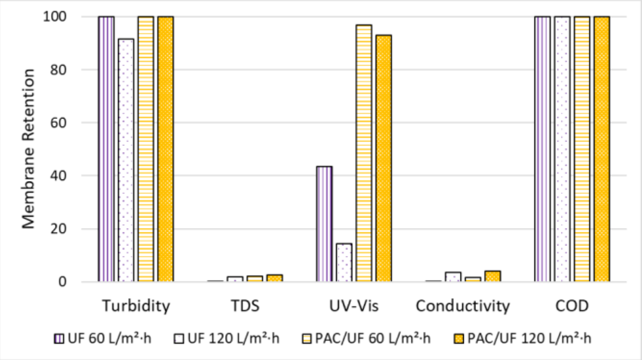

Figure 5. Membrane retention of UF and hybrid PAC/UF

An interesting finding was observed on UV-Vis removal (organic substance), the PAC/UF was able to reach significantly higher removal efficiency in the range of 93-96% compared to a single UF membrane within the range of 14-43%. The higher organic removal was also supported by the research by Elma et al. (2022) that used combination coagulation-PAC-MF membranes capable of removing UV254 by 94.66%. On the other hand, studies using commercial polysulfone membranes were only able to remove UV254 by 41% (Adams et al., 2014). High organic removal occurred due to the adsorption ability of PAC that increases the removal power of organic micro molecules.

Furthermore, insignificant efficiency on TDS and conductivity (<5%), which represent salinity in water, were observed for both experiments. Significantly lower removal of this parameter was caused due to salt ions, which are monovalent bonds and smaller than the average pore size of the PES membrane of 30 nm (loose membrane). Ion removal can be effectively carried out in RO, and some can be carried out by NF membranes (Madsen, 2014). TDS removal using UF/membrane prefiltration is only worth 7.7% compared to the prefiltration configuration UF/RO membrane, which can set aside a TDS of 87.46% ((Dagar et al., 2023)). The weak salt rejection in the UF membrane could occur due to the concentration polarization of foulant at the top layer of the membrane, causing salt ions diffusion that has the smallest size and molecular weight among other foulants such as organic molecules (Salinas-Rodríguez et al., 2021). However, another study revealed that PAC is able to remove a portion of TDS in seawater up to 67.9 % (Tomar & Rastogi, 2018).

3.3 Characterization of PES Ultrafiltration Membrane

The characterization of the fouled 30 nm UF PES membrane was investigated by measurement using a Scanning Electron Microscope (SEM) (Quanta FEG 650, FEI, USA). The morphology of the membrane was shown on an enlarged micrometer scale reading. SEM was carried out by taking images of the surface of the membrane that had gone through filtration experiments. In general, a good membrane is a membrane that has small pores in large numbers and is evenly distributed (Arahman, 2017). However, because the membrane has been carried out for filtration and is experiencing fouling, likely the membrane pores are not visible because they are blocked by foulant that can be seen in (Figure 6).

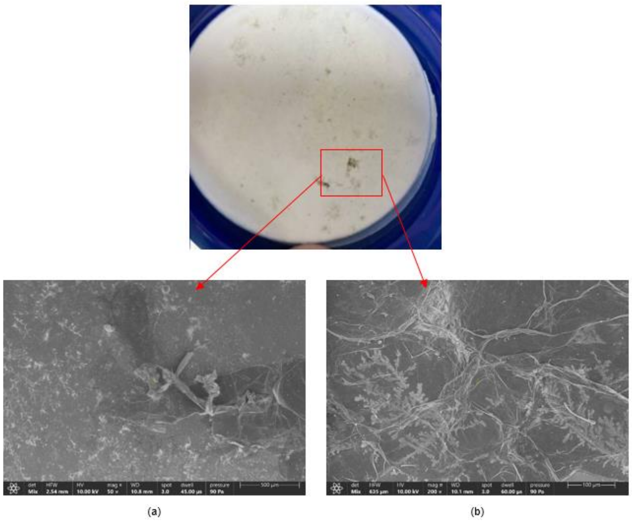
(a) (b)

Figure 6. Membrane surface SEM image (a) 50x and (b) 200x

Furthermore, membrane characterization was conducted by analyzing the EDS (Energy Dispersive X-Ray Spectroscopy), which provided component data for the two areas performed by SEM (See. Figure 6). The cake layer on the membrane has various constituent components in the form of organic and inorganic compounds and salt ions, where the largest percentage of atoms that make up the cake layer consists of carbon (C) (43-47%), oxygen (O) (24-35%), sodium or salt (Na) (7-13%), and chlorine (Cl) (7-13%) atoms (see Table 1). The atomic elements in region A were found to be different from region B. The difference indicates that the foulants on membrane surface was not be homogeneous distributed over the entire surface of the membrane (Salinas-Rodriguez S. G., 2021). This phenomenon can be attributed to the average pore size, contact angle, and material properties of UF PES 30 nm membrane to reject the foulant compound.

Table 1. Result of EDS analysis in elemental form

Elements	Atomic (%)	
	Region A	Region B
C	43.0	47.1
O	24.2	35.3
Na	13.7	7.0
Mg	0.9	0.8
Al	0.2	-
Si	1.6	-
P	0.3	-
S	3.0	2.0
Cl	13.1	7.8
N	-	-

3.4 Characterization of PES Ultrafiltration Membrane

The characterization of PAC that had been employed to run PAC/UF filtration was further conducted by Fourier Transform Infrared (FTIR) spectrophotometry (See Table 2). FTIR is a vibrational spectroscopic analysis method used to determine the functional groups to analyze organic and inorganic compounds found in activated carbon (Hayu et al., 2021). At wavelength 1987 cm^{-1} it was identified that there is a functional group C=C=C. This bond can provide an active adsorption site and allows molecules to interact with each other to help adsorb organic compounds. At wave number 1406 cm-1, an O–H functional group. At wave number 1017 cm-1, the C–O functional group was identified. The functional group of the PAC represents adsorption ability and was expected to improve the elimination of foulants that cause cake layers on the surface area of the membrane in the previous discussion as EDS results. As a result, the precoat PAC on the top of membrane could improve the quality of permeate water produced by the filtration process compared to the permeate quality by the UF membrane.

Table 2 Functional group of powdered activated carbon

Numb	Structure	Frequency Region (cm^{-1})
1	C=C=C strong	1987
2	O–H medium	1406
3	C–O medium to strong	1017
4	C–H strong	872
5	C–I strong	572

4. CONCLUSION

The lab scale investigation on the PAC/UF 30 nm PES operated at 60 and 120 $L/m^2 \cdot h$ revealed that the combined PAC/UF is a promising technology for SWRO pretreatment. In general, the UF and PAC/UF had high removal of turbidity and COD up to >90%. However, additional PAC resulted in higher removal of organic compounds in parameter UV-Vis up to 93-96%. On the other hand, insignificant removal for salts <5% was revealed for PAC/UF. Related to membrane performance, PAC/UF and UF revealed a better performance for combined PAC/UF considering thicker layer / higher membrane resistance due to additional PAC.

ACKNOWLEDGMENT

The authors express gratitude to “Kurita Asia Research Grant” from Kurita Water and Environment Foundation for the research funding. The authors are grateful to the North Jakarta SWRO Installation for providing outlet DAF water as a feed water for the experiment and the Laboratory of Sanitary and Environmental Engineering Universitas Indonesia for providing the sample analysis.

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