



Terakreditasi: SK No.: 60/E/KPT/2016 Website : http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 17 No. 1, Maret Tahun 2017, Hal. 43-52

Fouling Evaluation of Modified Cellulose Acetate Asymmetric Membranes for Various Brackish Water Treatment

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Abstract

Water treatment technology which is being widely developed is membrane technology. It is an alternative technology with sieving mechanism, electrodialysis, electro deionization, and reverse osmosis principle. The main purposes of this research are to investigate the effects of additives and thermal treatment on the cellulose acetate membrane morphology and performance in brackish water treatment. The membrane was fabricated by preparation of dope solution consits of cellulose acetate and acetone. The membrane was casted via dry-wet NIPS method using mechanic casting knife. Dope solution was prepared by varying the PEG concentration of 1 and 5wt-% and the annealing temperature at 60 $^{\circ}$ and 25 $^{\circ}$ for 10 seconds. The backwash technique was used to remove foulant deposition on the membrane surface. The results of SEM and FTIR showed that the higher PEG addition to the dope solution resulted in larger membrane pore size and increase the hydrophilic properties of membrane. However the higher temperature and the longer annealing time, the skin layer of membrane become denser. The result was also shown that back wash technique could reduce foulant deposition in the nanofiltration membrane.

Keywords: additive; back wash; brackish water; cellulose acetate; polyethylene glycol; thermal annealing

Abstrak

EVALUASI FOULING PADA ASIMETRIK MEMBRAN SELULOSA ASETAT TERMODIFIKASI UNTUK PENGOLAHAN BERBAGAI SUMBER AIR PAYAU. Teknologi pengolahan air yang saat ini sedang dikembangkan secara luas adalah teknologi membran. Membran merupakan teknologi alternatif yang menggunakan prinsip mekanisme sieving, elektrodialisis, elektrodeionisasi, dan reverse osmosis. Tujuan utama penelitian ini adalah untuk mengkaji pengaruh dari penambahan aditif dan perlakuan panas pada membran selulosa asetat terhadap morfologi dan kinerja membran dalam mengolah air payau. Membran dibuat dengan menyiapkan larutan dope yang terdiri dari selulosa asetat dan aseton. Membran dicetak menggunakan metode dry-wet NISP dengan alat pisau casting manual. Larutan dope dibuat dengan memvariasi konsentrasi PEG 1 dan 5 %b/b serta variasi suhu annealing yaitu pada 60 $^{\circ}$ C dan 25 $^{\circ}$ C selama 10 detik. Teknik pencucian balik digunakan untuk menghilangkan foulant yang menempel di permukaan membran. Hasil analisa SEM dan FTIR menunjukkan bahwa semakin besar konsentrasi PEG dalam larutan dope akan menghasilkan membran dengan ukuran pori yang lebih besar dan meningkatkan sifat hidrofilik membran. Namun dengan semakin tinggi suhu annealing dan semakin lama waktu annealing akan menghasilkan membran dengan skin-layer yang lebih rapat. Hasil penelitian juga menunjukkan bahwa pencucian balik dapat mengurangi jumlah foulant yang menempel pada membran nanofiltrasi.

Kata kunci: aditif; pencucian balik; air payau; selulosa asetat; polietilen glikol; thermal annealing

How to Cite This Article: Kusworo, T.D., Budiyono, Sofiana, E., Rochyani, U.N.A., and Utomo, D.P., (2017), Fouling Evaluation of Modified Cellulose Acetate Asymmetric Membranes for Various Brackish Water Treatment, Reaktor, 17(1), 43-52, http://dx.doi.org/10.14710/reaktor.17.1.43-52.

INTRODUCTION

The alternative technology that is being developed in water treatment is membrane based separation (Baker, 2004; Mulder, 1996). The membrane based technology does not require chemical additives when it is applied in water treatment unlike the existing conventional technologies (Pendergast and Hoek, 2011; Baker, 2004). Moreover, membrane technology is compact so that the capital investment cost is lower than existing technologies, beside that the membrane is modular that means easy to be scaled up (Baghbanzadeh et al., 2015; Nicolaisen, 2003). The investigation which is focused on membrane fabrication process condition is still interesting to be investigated. This is due to many factors that affect on fabricated membrane. (Baker, 2004).

One of the membrane materials that commonly used in membrane fabrication for water treatment is cellulose acetate (CA) (Sivakumar et al., 2006). CA membrane is fabricated using Non-solvent Induced Phase Separation (NIPS) method where the polymer is converted from liquid phase to solid phase by the precipitation in non solvent immersion (Raharianto et al., 2007; Sagiv et al., 2008). The additives are often added to the dope polymer solution for a certain purposes. One of the several additives that is widely added to increase the permeability of membrane is polyethylene glycol (PEG). The addition of PEG in the membrane fabrication was known has the effect of increasing the permeate water flux because PEG is known as a porogent (pore-forming) organic substance on the membrane fabrication (Katsoufidou et al., 2005).

Morphological structure of membrane plays an important role in membrane separation behavior which determines the success of separation process (Mulder, 1996). The flux and rejection of certain compound are two parameters that expressed the performance of membrane separation. Membrane with high flux and rejection characteristics is favorable. However, the current condition in membrane material development will be found that the effort in increasing membrane flux will reduce the rejection performance of membrane and vice versa (Kusworo et al., 2017). The fouling in the membrane also becomes main problem in membrane separation process. The addtion of PEG in the membrane material is considered to increase the hydrophilicity properties of membrane which implies on membrane with anti-fouling behaviour (Kusworo et al., 2017; Ahmad, 2005).

The prost treatment of membrane also gives an significant effect to membrane structure. Thermal treatment on the membrane influences the membrane flux and rejection (Kusworo *et al.*, 2015). The variation of thermal annealing temperature to membrane materials affects on membrane pores

structure. Annealing treatment on the membrane at temperature close to the glass transition temperature (Tg) is resulting a lower permeate flux and higher rejection compared with un-annealed membrane (Kusworo *et al.*, 2015; Mulder, 1996). The effects of PEG addition in dope solution and thermal annealing treatment on the CA membrane performance for brackish water treatment are investigated in this study. To our knowledge, there is no documentation on the use of combination PEG and thermal annealing process and backwash system to improve the stability membrane for brackish water treatment.

Therefore, the main objective of the present study was investigated the effect of backwash system to control the fouling formation on the brackish water treatment. The present study was also studied the effect of backwash duration and interval filtration on a productivity and selectivity. The present paper also examined the effects of flow rate on the backwash processes.

MATERIAL AND METHOD Materials

Cellulose acetate (CA) was purchased from MKR chemicals for membrane material. Acetone 99.75% from mallinckrodt chemicals was used as solvent. Ultra-pure distilled water from Integrated Laboratory of Diponegoro University was used as non-solvent. Polyethylene glycol (PEG) with molecular weight of 4,000 g.mol⁻¹ and original brackish water sample from coastal area of different cities: Demak, Jepara, and Semarang Indonesia.

Cellulose Acetate Membrane Characterization

Permeate water flux was determined by applying the membrane on filtration cell module as shown in Figure 1. Distilled water was streamed into the filtration cell and the operating pressure was maintained at 3 atm. Compacting process was performed for 30-45 minutes in order the polymer chains arrange themselves. After the compaction process, distilled water in the filtration cell is replaced with brackish water sample. Permeate water flux measurement was performed by measuring the volumetric flowrate of permeate water that can fit over a certain time interval with intervals of 30 minutes. Flux values are calculated by the ratio of the volume of permeate per unit membrane area per unit time. Rejection efficiency was performed by determining the concentration of total dissolved solid, Ca²⁺, Mg²⁺, and turbidity of brackish water both of upstream and downstream. For every completed filtration process, the membrane was washed using backwash method. Backwash method aims to reduce the fouling of the membrane. Filtration process was initially performed for 30 minutes and 2 hours, after the filtration process, the membrane was backwashed using deionized water

for 1 minute, then it was calculated for the value of flux and rejection. After the membrane cleaning process, the cleaned membrane was reused for produced water to obtain the separation peprformance of cleaned membrane. The value of flux and rejection were calculated using the following equation:

$$J_w = \frac{V_w}{A.t.P} \tag{1}$$

 $J_w = Flux (L.m^{-2}.h^{-1} bar^{-1})$

- V_w = Volume of Permeate (liter)
- A = Membrane Surface Area (m^2)
- T = Time (hour)
- P = Pressure (bar)



Figure 1. Membrane filtration system: (1) feed ; (2) membrane ; (3) retentate ; (4) permeate

The morphology of membrane structures were determined using a Scanning Electron Microscopy (SEM). The fabricated CA membrane samples were fractured in liquid nitrogen. The membranes were mounted on an aluminium disk with double surface tape and then the sample holder was placed and evacuated in a sputter-coater with gold. Through this analysis, it can be seen the cross-sectional and the surface morphology of the membrane with a certain magnification.

RESULTS AND DISCUSSIONS

The Effect of PEG Additive on the Separation Performance with Various Brackish Waters

The data of PEG additive effects on membrane separation performance were presented in Figure 2. The Figure 2 displayed that the higher PEG concentration in the polymeric dope solution, the permeate water flux increased significantly. The enhancement of flux could be due to more poers were formed during fabrication process. The addition of PEG concentration of 5% wt on dope solution affects on the pores formation. Membrane pore formed by the more or greater with the addition of PEG concentration. PEG is an additive that not only acts as a pore-forming agent or multiply the number of pores but also led to an adjustment movement of molecules in the formation of cellulose acetate membranes. This is because PEG is a hydrophilic additive, so that an

increase in the concentration of PEG induced the macro-voids formation in membrane skin layer (Idris and Yet, 2003; Liu et al., 2017). As a result, the larger of pore size of the membrane will increase the permeate water flux of the membrane. The various brackish water sources also caused the varying in permeate water fluxes. For example, based on the Figure 2 can be seen that brackish water from Semarang has the higher permeate flux, followed by brackish water from Jepara and Demak. These could be due to the organic and mineral solute content in the brackish water. Brackish water from Semarang may contains the lowest organic and mineral solute than brackish water from Jepara and Demak. Based on this result, generally the permeate water flux is influenced by the characteristic of feed water.



Figure 2. The effect of PEG addition on the permeate water flux for various brackish water feed: (a) water of Jepara; (b) water of Semarang; (c) water of Demak

As depicted in Figure 3 for brackish water from jepara, Figure 4 for brackish water from Semarang, Figure 5 for brackish water from Demak. It can be

seen that higher PEG loading in polymeric dope solution, the membrane rejection values for Ca²⁺ and Mg²⁺ decreases. Membrane with a concentration of 1 wt% PEG resulted in rejection values greater than the membranes with PEG concentration of 5 wt%. This might be due to the higher concentration of PEG in the dope solution will increase the number pores and pore size. PEG acts as a pore-forming agent (porogent) which is greatly soluble in water so that the PEG molecules diffuse into the coagulation bath containing water and leave pores in the CA membrane matrix (Haddad et al., 2004). As a result, the greater the concentration of PEG is added, will form macro-voids are greater in the membrane, the greater porosity and pore size of the membrane will cause a lot of monovalents and divalents ions passed through membrane barrier, so that the value of rejection is getting smaller which causes membrane's ability to filter the Ca^{2+} and Mg^{2+} is reduced. At least the addition of PEG in the membrane matrix is to improve the performance of the membranes in term of selectivity, especially Ca^{2+} and Mg^{2+} rejection.

Based on the experimental result, the rejection of Ca^{2+} is higher than Mg^{2+} . It might be due to the mechanism of membrane transport is dominated by sieving mechanism where the selectivity of membrane depends on solute molecular weight which is related to the molecular size. In this case, the affinity of Ca^{2+} and Mg^{2+} are similar because they are in the same group of metals. But Ca^{2+} is larger in molecular size than Mg^{2+} which is showed by their molecular weight of 40 g.mol⁻¹ and 24 g.mol⁻¹, respectively. This would cause the rejection of Ca^{2+} is higher than Mg^{2+} .



Figure 5. The effect addition of PEG additive on the rejection of Ca²⁺ and Mg²⁺ for brackish water from Demak

The Effect of Thermal Annealing on Membrane Stability

In this research involved four variations of the membrane, the membrane 1 with a concentration of 1 wt% PEG and without annealing, membrane 2 with a concentration of 5 wt% PEG and without annealing, the membrane 3 with 1 wt% PEG concentration with annealing temperature of 60° C, and the membrane 4 with 5 wt% PEG concentration and annealing temperature of 60°C.



Figure 6. The effect of annealing treatment on the flux at various membrane and brackish water sources: (a) water from Jepara; (b) water from Semarang; (c) water from Demak.

In accordance with Figure 6, the figure illustrated the comparison of membrane flux values without annealing and with annealing at 60°C on each brackish water source (Jepara, Semarang and Demak). Judging from the overall value of the flux at each source, flux values by using a membrane with annealing temperature of 60°C tends to produce a stable water flux rather than the membrane without annealing treatment. The thermal treatment on the membrane allowed the polymer molecules becomes more crystalline (Han and Bhattacharya, 1995). As a result of membrane pore narrowing is the lowe permeate water flux of the membrane.

The variation of brackish water sources are related to the variation of Ca^{2+} and Mg^{2+} rejection. Overall, the rejection of Ca^{2+} is higher than Mg^{2+} due to the different molecular size between Ca and Mg. The rejection of solute is influenced by the concentration of solute in the feed water. The higher concentration of solute in feed water will result in the higher solute rejection. Brackish water from Demak has the highest average solute rejection, followed by brackish water from Jepara and Semarang. It strengthened the pervious statement that brackish water from Semarang has the lowest solute concentration than Jepara and Demak.

Figure 7 showed the value of rejection Ca^{2+} of membrane without annealing and with annealing at 60°C for each brackish water source (Jepara, Semarang and Demak). Overall the rejection values of Ca^{2+} by using a membrane with a temperature of 60°C thermal annealing tends to produce a more stable and higher rejection rate for Ca²⁺ than the membrane without using thermal annealing. The post-treatment of thermal annealing in this study improved the performance of the membrane in terms of solute rejection due to the more dense membrane formation induced by thermal treatment. The higher annealing temperature and the longer annealing time on the membrane induced polymer shrinking so that the membrane becomes more dense (Ahmad, 2005). As a result of narrowing of the membrane pores is the increase of membrane rejection performance.





Figure 7. The effect of annealing treatment on the rejection of Ca²⁺ with various membrane and brackish water sources (a) water of Jepara; (b) water of Semarang; (c) water of Demak

The Effect of Backwash Processes on the Flux and Ca^{2+} and Mg^{2+} Rejection

The effects of backwash processes on the flux and Ca²⁺, Mg²⁺ rejection were depicted in Table 1. In Table 1 can be seen that the flux after backwash processes was increased with the time of backwash process. However at 2 minutes time of backwash, the flux decreased insignificantly. This phenomenon indicated that the fouling was rised on membrane surface. In Table 1 is shown the data of Ca²⁺ and Mg²⁺ rejection respectively. The rejection value was also increase. This phenomenon indicated that the performance of cellulose acetate membranes with 5 wt% and 60°C for 10 seconds. Fouling formed by the ions in the feed water that settles and blocked the process of filtration. In a certain time which resulted in flux values raises is possible damage to the membrane and the pressure on the tool suddenly becomes bigger. Backwash technique is effective in removing the foulant from the membrane and can restore the initial flux values approaching flux at the beginning of the filtration process (Sagiv et al., 2008).

Table 1. Membrane with 5 wt% PEG and 60°C during
10 seconds

		10	seconds.		
Back- wash	Flux ^a	Ca ^b Rejection	Incresing of Ca ^c Rejection	Mg ^b Rejection	Incresing of Mg ^b Rejection
1	125.01	55.56	5.94	20.35	13.04
2	116.44	55.85	4.60	18.67	12.45
5	128.22	44.74	3.72	30.09	9.60
7	133.24	48.83	3.24	25.15	17.01

^a (Lh⁻¹m⁻²bar⁻¹) ^b(ppm) ^c(%)

According to the other researcher, the fouling on the membrane generally causes a decrease in solvent flux across the membrane. There are two processes associated with the fouling phenomenon, which internally will be the intoxication (poisoning) and externally is growth of fouling itself (Haddad *et al.* 2004). In particular membrane processes, such as reverse osmosis, membrane density and the increase in osmotic pressure on the membrane surface can also lead to decreased solvent flux across the membrane, but this effect can not be categorized as fouling. From the experimental observations using brackish water as feed solution, it can be estimated that the major cause of declining flux foulant layer on the membrane wall, even though other factors also can not be ignored.



Figure 8. Influence of water sources on (a) rejection of Ca²⁺ and (b) rejection of Mg²⁺ after being backwashed for various time

In Figure 8, the graphic of Ca^{2+} and Mg^{2+} rejection decreased and the increase during the filtration process takes place. The longer the operating time will increase the tendency of rejection although relatively small, it is likely caused by foulan attached or bound to the membrane which would be a new filter for the water feed. From the rejection of Ca^{2+} and Mg^{2+} , Ca^{2+} ions more easily than rejection of Mg^{2+} ions (Katsoufidou *et al.*, 2005).

Comparison the Effects of Backwash on the Filtration Processes for 30 Minutes and 2 Hours

To determine the effectiveness of the backwash for eliminating the fouling of the membrane, then use the variable filtration time, 30 minutes and 2 hours.

In Table 2 can be seen the value at the time of filtration flux for 2 hours and then in the backwash for 1 minute to have a bigger value than at the time of filtration for 30 minutes later in the backwash for 1 minute, and when the backwash flux value is bigger than the current filtration. The longer time goes by filtration, the more foulan that clog the pores of the membrane, so that the permeate flow is becoming slower (Sagiv et al., 2008). At the time of the backwash process is done, the results of filtration permeate is passed back through the membrane and pore clogging foulan that had come with the permeate flow and be activated cake on the membrane surface. the particles can be cleaned with the backwash process, this is called reversible fouling, which is why flux value at the time of backwash is bigger than at the time of filtration (Ye et al., 2003; Katsoufidou et al., 2005).

In Table 2 presented value of Ca²⁺ and Mg²⁺ rejection. Rejection of Ca^{2+} and Mg^{2+} at 30 min filtration time has a bigger value than the filtration time 2 hours. Rejection value in generally inversely proportional to the value of the flux, and flux value is inversely proportional to time. Overall, same as the permeate flux, rejection of Ca^{2+} and Mg^{2+} during the backwash process has a higher value than during the filtration process. At the time of the filtration process, the longer time of operation, the smaller the flux due to the blockage the pores of the membrane, which resulted in fouling that affect the rejection of Ca^{2+} and Mg²⁺. Fouling occurs due to the highly specific interactions between the physical and chemical dissolved solids with a membrane (Liu et al., 2017). The more fouling on the membrane, it means the salt content in the feed water and only retained a lot of salt with a diameter smaller than the membrane pores who can qualify, so the rejection of Ca^{2+} and Mg^{2+} is also getting bigger. Rejection of Ca^{2+} and Mg^{2+} after backwash process has increased compared to the previous filtration process. The molecules that exist in the active layer of the membrane, the result of the accumulation at the time of filtration is done, will be eliminated by doing the backwash process, so that the rejection of Ca2+ and Mg2+ on permeate has a bigger value (Liu et al., 2017).

Table 2. Data of flux and Ca ²⁺ , Mg ²⁺ rejection at
backwash time variable 1 minute and filtration time 30
minute and 2 hours

Data	Filtration 30 Minute, Backwash 1 Minute				
2	1 st hour	2 nd hours	3rd hours		
Filtration Time					
Flux (L.h-1m-2bar-1)	139,278	181,741	209.767		
Ca Rejection (%)	34,211	40,789	47,369		
Mg Rejection (%)	9,722	11,389	19,444		
Backwash Time					
Flux (L.h ⁻¹ m ⁻² bar ⁻¹)	10,021	13,248	17,155		
Ca Rejection (%)	35	42,632	50		
Mg Rejection (%)	19,694	15,139	22,222		

Data	Filtration 2 hours, Backwash 1 Minute				
Duiu	1 st hour	2nd hours	3rd hours		
Filtration Time					
Flux (L.h ⁻¹ m ⁻² bar ⁻¹)	655,626	655,626	655,626		
Ca Rejection (%)	31,579	31,579	31,579		
Mg Rejection (%)	8,333	8,333	8,333		
Backwash Time					
Flux (L.h ⁻¹ m ⁻² bar ⁻¹)	12,569	12,569	12,569		
Ca Rejection (%)	36,842	36,842	36,842		
Mg Rejection (%)	11,111	11,111	11,111		

Characterization of Cellulose Acetate Membranes for Analytical Scanning Electron Microscopy (SEM)

Analysis of SEM (Scanning Electron Microscopy) can be used to determine the structure of membrane morphology. The results of this test in the form of photos and transverse membrane surface appearance using electron microscopy (Mulder, 1996). In this study, SEM analysis to determine the structure of membrane morphology is formed. The membranes used in this SEM test is membranes with PEG concentration of 5% and a heating temperature of 60°C, with brackish water from Demak. And in this study, to determine the morphological characterization of the membrane can be seen in Figure 9.

Figure 9 shows the surface and cross section of acetate membranes with cellulose variable magnification of 2500 times with the addition of 5 wt% PEG and heating temperature of 60°C. Based on cross-sectional images of the membrane surface it appears that the cross-section, appears cellulose acetate membrane that forms an asymmetric membrane, as forming at least two layers of the membrane. Layer formed on the membrane is dense layer, an intermediate layer and porous substucture. Asymmetric membrane is a membrane with pore size of the outer side more dense with a thickness between 0.1-0.5 lm, while the pore size in the lighter side with a thickness between 50-200 lm (Mulder, 1996).



Figure 9. Surface (a) and cross section (b) cellulose acetate membrane

Membrane surface and membrane pore was very smooth or cavity smaller. This suggests the addition of PEG as an additive function of the membrane is intended to enlarge the pore membrane with the membrane while maintaining resistance against external factors (Chou *et al.*, 2005). In this case, the additive (in this study is PEG) initially fills the cellulose acetate matrix. Furthermore, in coagulation with non-solvent, PEG was leached by water and formed voids of pores in the membrane. The thermal treatment which was given to the membrane material enhanced the movement of polymer molecules to form more crystaline material.



(a)

In addition, thermal treatment also decreased the unselective macro-voids formed in the membrane fabrication, due to the increased molecular movement within the membrane (Baker, 2004). The less amount of unselective macro-voids in the membrane results in the smaller pores or cavities are formed, so that the pores of membrane was closer each other.

Morphology of Cellulose Acetate Membranes for Analytical Scanning Electron Microscopy (SEM) After Filtration and Backwash

To determine the effect of backwash on the structure of the resulting membrane morphology, using analysis of SEM (Scanning Electron Microscopy). Backwash of the membrane was carried out at a vulnerable time backwash 1, 2, 5, and 7 minutes, while the membrane was analysed using SEM cellulose acetate membrane 5 wt%, the heating temperature of 60°C for 10 seconds, and backwash for 1 minute.

Figure 10 is a cross-sectional images on the surface of the cellulose acetate membrane filtration process for 6 hours and after backwash. At 6 hours of filtration membranes seen in the picture, there are many foulan that clog the pores of the membrane and deposited on the membrane surface, resulting increasingly smaller flux and rejection Ca^{2+} and Mg^{2+} were bigger during filtration.

Figure 11 is a cross section on the cellulose acetate membrane filtration process for 6 hours and after backwash. Can be seen in the picture, there are so many foulan that settles on the surface of the membrane and foulan that clog pores or cavities membrane. As explained in the previous discussion, it is caused by the accumulation of the molecules in the active layer during the membrane filtration process that is performed before the washing process (Elimelech *et al.*, 1997; Liu *et al.*, 2017). Fouling can be controlled with regular membrane washing (Katsoufidou *et al.*, 2005; Katsoufidou *et al.*, 2007). In filtration experiments using brackish water sourced from Demak, has done washing (backwash) of the membrane.



Figure 10. Surface section after filtration (a) and after backwash (b)



(a) (b) Figure 11. Cross section after filtration (a) and after backwash (b)

Rejection of Ca^{2+} and Mg^{2+} in the leaching process is an increase compared to during the process of filtration. This is because the feed water washing process that passes through the membrane carries most of the ions Ca^{2+} and Mg^{2+} build up/clogging the pores of the membrane, so that the permeate during the washing process contains Ca^{2+} and Mg^{2+} is more than that permeate during the filtration process.

CONCLUSION

Backwash method or membrane washing is effective for cleaning the membrane from fouling, but backwash system can't restore the flow rate returned to normal. The molecules exist in the active layer of the membrane results accumulated during filtration was lost, so the rejection of Ca^{2+} and Mg^{2+} on permeate has a bigger value. The smaller value of flux and rejection are bigger value indicates that the membrane performance is good and stable. In this research was also conducted comparison of flux and rejection methods backwash filtration time of 30 minutes and 2 hours. Value of flux and rejection at the time of 2 hours has a bigger value than 30 minutes. Backwash operating time and interval of filtration affects on productivity and selectivity. The results of SEM analysis displayed that the resulting CA membrane is an asymmetric membrane. Foulant deposition on the membrane surface is accumulated during the filtration process, while at the time of backwash processes (leaching), membrane fouling has decrease.

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