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International Conference on Chemistry and Material Science (IC2MS) 2017

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The INTERNATIONAL CONFERENCE ON CHEMISTRY AND MATERIAL SCIENCE (IC2MS) 2017

“Advancing to the frontier of innovation in chemistry and material science”

4-5 November 2017

IJEN SUITES RESORT & CONVENTION

MALANG, INDONESIA



PREFACE

On behalf of the Organizing Committee, I would like to welcome all of you, keynote speakers, invited speakers, distinguished guests, and participants, to the **International Conference on Chemistry and Material Science (IC2MS) 2017**. This conference is organized by Chemistry Department, Brawijaya University, and is held for the first time. The IC2MS runs for 2 days, 4-5 November 2017, and consists of 5 mini symposia, they are:

- Analytical and Inorganic Chemistry
- Biochemistry and Organic Chemistry
- Synthetic and Catalyst Materials, Renewable Energy and Fuel Cells, Molecular Science & Computational Chemistry
- Materials Characterization
- Nanomaterials & Nanodevices and Physical Chemistry

The accepted papers of the conference will be published in IOP Conference Series: Materials Science and Engineering. Around 200 people are attending this conference. They consist of 150 presenting participants, 50 non-presenting participants, 5 keynote speakers, and 6 invited speakers. In terms of country of origin, the participants of the IC2MS are coming from 8 countries, including Indonesia, Brunei Darussalam, Thailand, Taiwan, Japan, Kazakhstan, Australia, and Singapore.

The IC2MS should bring advantages for all participants. The IC2MS attendees can exchange and share their experiences and research results on all aspects of chemistry and material sciences. The IC2MS also provides a premier interdisciplinary platform for researchers, practitioners and educators to present and discuss the most recent innovations, trends, and concerns as well as practical challenges encountered and solutions adopted in the fields of chemistry and material sciences.

The ICM2S could not become a reality without the help and assistance of many parties. Thus, in this occasion I would like to sincerely thank the Rector of Brawijaya University, Dean of Faculty of Mathematics and Natural Sciences Brawijaya University, Head of Chemistry Department, Brawijaya University, all members of the Organizing Committee, Ijen Suites Hotel, and all sponsors, who have provided meaningful help and assistance for the implementation of this conference.

Malang, 4 November 2017

Chairman of the Organizing Committee

Anna Safitri, Ph.D

CONFERENCE PHOTOGRAPHS



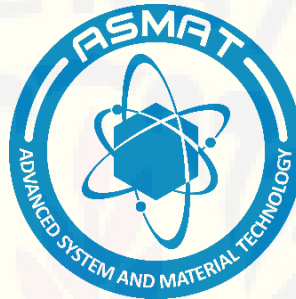
FUNDING ACKNOWLEDGEMENT

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Peer review statement

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Effect of Evaporation Time on Separation Performance of Polysulfone/Cellulose Acetate (PSF/CA) Membrane

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Effect of Evaporation Time on Separation Performance of Polysulfone/Cellulose Acetate (PSF/CA) Membrane

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Abstract. Polysulfone and cellulose acetate are common material in separation. In this research, polysulfone/cellulose acetate (PSF/CA) blend membrane was prepared. The aim of this research was to study effect of evaporation time in casting of PSF/CA membrane and its performance in filtration. CA was obtained by acetylation process of bacterial cellulose (BC) from fermentation of coconut water. Fourier Transform Infra Red (FTIR) Spectroscopy was used to examine functional groups of BC, CA and commercial cellulose acetate. Substitution of acetyl groups determined by titration method. Blend membranes were prepared through phase inversion technique in which composition of PSF/PEG/CA/NMP(%w) was 15/5/5/75. Polyethyleneglycol (PEG) and N-methyl-2-pyrrolidone (NMP) were act as pore forming agent and solvent, respectively. Variation of evaporation times were used as parameter to examine water uptake, flux, and morphology of PSF/CA blend membranes. FTIR spectra of CA show characteristic peak of acetyl group at 1220 cm^{-1} indicated that BC was acetylated successfully. Degree of substitution of BCA was found at 2.62. Highest water flux was performed at 2 bar obtained at 106.31 $\text{L.m}^{-2}.\text{h}^{-1}$ at 0 minute variation, and decrease as increasing evaporation time. Morphology of PSF/BCA blend membranes were investigated by Scanning Electron Microscopy (SEM) showed that porous asymmetric membrane were formed.

1. Introduction

Ultrafiltration technology has been interesting due to effectivities in separation performance. Polysulfone is common material for ultrafiltration. It has high durability and stability under mechanical, thermal and chemical treatment [1]. Unfortunately, it strongly hydrophobic, thus susceptible to fouling. Fouling in membrane was a major problem in environmental application, which cause lower water flux and membrane life, thus higher energy in operation [2]. Many research has been taken in order to improve performace of polysulfone, they are impregnating PSF with nanoparticles such as silver, TiO_2 , ZrO_2 , SiO_2 [3][4] or blending with hydrophilic polymers such as nanocellulose [5] and cellulose acetate [6].

Blend polysulfone to another hydrophilic polymer is a simple way to improve its performance. Cellulose acetate known as good hydrophilic ultrafiltration materials, but has low stability in chemical treatment. Adding cellulose acetate in small portion to polysulfone, improve its properties, and reduce fouling properties. Cellulose acetate is able to obtained from natural resources such as refined fiber, cellulosic waste, deinked newspaper, cotton linter pulp and bacterial cellulose [7][8][9][10]. Bacterial



cellulose is one of cellulose material with high crystallinity, and purity. They bring advantages in reduce step in isolation of cellulose, such as delignification and bleaching [10].

Polymer blend membranes can be prepared by phase inversion technique. Polymer solution is poured into glass plate then immerse into water in coagulation bath [6][11][12][13]. There are many factors affecting characteristic of blend membranes such as coagulation bath temperature [11][13], post treatment after solidification [12], and evaporation time [14] before immersion into coagulation bath. Variation on evaporation time has different characteristic due to evaporation of solvent before immersion into non solvent.

In this research, PSF/CA blend membranes are prepared by phase inversion technique. In the present work, cellulose acetate from renewable resources are used. Bacterial cellulose are used as the source for cellulose acetate (CA). Evaporation time is used as parameter to study characteristic of membrane and separation performance on filtration of oil/water emulsion.

2. Materials and Methods

Food grade sugar and coconut water were obtained from traditional market. *Acetobacter xylinum* was obtained from biology laboratory of Universitas Negeri Semarang, glacial acetic acid, sodium hydroxide, n-methyl-2-pyrrolidone (NMP), polyethyleneglycol-400 (PEG-400), sulfuric acid, acetic anhydride were obtained from Merck. Polysulfone (MW~35.000) and commercial cellulose acetate (MW~30.000) were obtained from Sigma Aldrich.

2.1. Preparation of Cellulose Acetate

Bacterial cellulose acetate was prepared by acetylation of bacterial cellulose. Bacterial cellulose was produced by fermentation of *Acetobacter xylinum* in coconut water medium [10]. Acetylation of bacterial cellulose was using acetic anhydride as precursor and sulfuric acid as catalyst [7][8]. Brown solution was obtained after acetylation at 60 °C for 4 hours. The final product was filtered and washed until neutral pH and then dried at 50 °C. The degree of substitution was determined by titration method [8]. Functional groups of bacterial cellulose and bacterial cellulose acetate were investigated using Fourier Transform Infra Red (FTIR) Spectroscopy. Thermal characteristic of cellulose acetate was examined by Differential Scanning Calorimetry (DSC 8230- Rigaku) analysis by determine Transition Glass Temperature (T_g), Melting Temperature (T_m), Entalphi of fusion on crystalline region (ΔH_f) and crystallinity index (% X_c) which determined by equation (1)[15]. Fusion entalphi of perfectly crystalline region of cellulose triacetate (ΔH_{100}) was 58.8 J.g⁻¹ taken from[16]. Thermal analysis were carried out from 30°C to 350 °C with heating rate at 5 °C/minutes. Furthermore, cellulose acetate was used for membrane preparation.

$$X_c(\%) = \left(\frac{\Delta H_f}{\Delta H_{100}} \right) \times 100 \dots \dots \dots (1)$$

2.2 Preparation of PSF/CA blend membranes

Blend membranes was prepared through phase inversion technique. Polysulfone (PSF) was blended with Cellulose Acetate (CA) using Polyethyleneglycol-400 (PEG) and N-Methyl-2-Pyrrolidone (NMP) as an aditive and solvent, respectively. Composition of PSF/BCA/PEG/NMP were 15/5/5/75 (%w). Evaporation times (0; 0.5 ; and 1 minutes) were use as parameter to study separation performance of blend membranes. Morphology of blend membranes was examined by Scanning Electron Microscopy (SEM) at Politeknik Negeri Jember. Porosity of PSF/CA membranes were calculated by equation (2), with density of water (ρ) was 998 kg/m³, membrane area (A) and membrane thickness (l). Weight of dry (w_{dry}) and wet (w_{wet}) membranes were obtained by immersion dry membrane on water for 24 hours. Surface of the membrane was wiped out to remove excess water, then weight as wet weight [19].

$$Porosity (\%) = \frac{w_{wet} - w_{dry}}{\rho \cdot A \cdot l} \times 100 \dots\dots\dots(2)$$

2.3 Separation Performance of PSF/CA blend membranes

Blend membranes performance was examined using dead-end ultrafiltration cell at 200 kPa in pressure[17]. Water flux (J) was determined by permeation of pure water flux (eq.3), where V was defined as volume of water passed through membrane (L), A was membrane area (m²) and Δt was sampling time for permeation of pure water.

$$J = \frac{V}{A \cdot \Delta t} \dots\dots\dots(3)$$

Separation performace of blend membrane was studied by filtration of dichloromethane in water emulsion. Dichloromethane in water was prepared as previous research [18]. Fouling character was evaluated by Flux Recovery Ratio (FRR), Total Fouling (R_t), Reversible Fouling (R_r) and Irreversible Fouling (R_{ir}). FRR was determined by equation (4), which J_{w1} was pure water flux and J_{w2} was recovered water flux after filtration. Total, Reversible, and Irreversible Fouling were determined by equation (5), (6) and (7), respectively, with J_p was permeate flux[2][19].

$$FRR (\%) = \left(\frac{J_{w1}}{J_{w2}} \right) \times 100 \dots\dots\dots(4)$$

$$R_t (\%) = \left(\frac{J_{w1} - J_p}{J_{w1}} \right) \times 100 \dots\dots\dots(5)$$

$$R_r (\%) = \left(\frac{J_{w2} - J_p}{J_{w1}} \right) \times 100 \dots\dots\dots(6)$$

$$R_{ir} (\%) = \left(\frac{J_{w1} - J_{w2}}{J_{w1}} \right) \times 100 \dots\dots\dots(7)$$

3. Results and Discussion

3.1 Characterization of Cellulose Acetate

Cellulose acetate has successfully synthesized from bacterial cellulose (BC). BC was obtained after fermentation of *Acetobacter xylinum* on coconut water. White pellicle was obtained after six days of fermentation. It has large amount of hydroxyl groups which interact each other through intermolecular hydrogen interaction. Therefore, BC was difficult to dissolve in various solvent.

Fourier Transform Infra Red (FTIR) spectra (Fig.1a) showed that BC had large amount of hydroxyl groups indicated by broad spectra at 3336.07 cm⁻¹, hence, BC was naturally hydrophilic which indicated by sharp peak of H-O-H bending from absorbed water at 1649.69 cm⁻¹. Acetylation process by acetic anhydride had substituted hydroxyl groups of BC with acetyl groups, which indicated by reduction intensity of -OH hydroxyl grups at 3573.36 cm⁻¹ leads reduced intensity of H-O-H bending at 1640.46 cm⁻¹. Presence of acetyl groups were showed by characteristic peak of C-O acetyl at 1223.35 cm⁻¹ and increased peak at 1742.18 cm⁻¹ indicating stretching of C=O esters. Shift peak at 887 cm⁻¹ to 903.67 cm⁻¹ showed vibration of β-glucosidic linkages between sugar unit. Detailed of FTIR spectra interpretation were showed at Table 1.

Table 1 FTIR spectra of bacterial cellulose and cellulose acetate

Wavenumber(cm^{-1})		Functional Groups [8][15]
Bacterial Cellulose	Cellulose Acetate	
3336.07	3573.36	O-H stretching
	2954.67	C-H stretching
2922.55		
1752.04	1742.18	C=O stretching of ester
1639.69	1640.45	H-O-H bending of absorbed water
1330.74	1372.23	C-H deformation
-	1223.35	C-O stretching of acetyl group
1058.96	1041.85	C-O asymmetric stretching of primary alcohol
887.00	903.67	β -glucosidic linkages between sugar unit

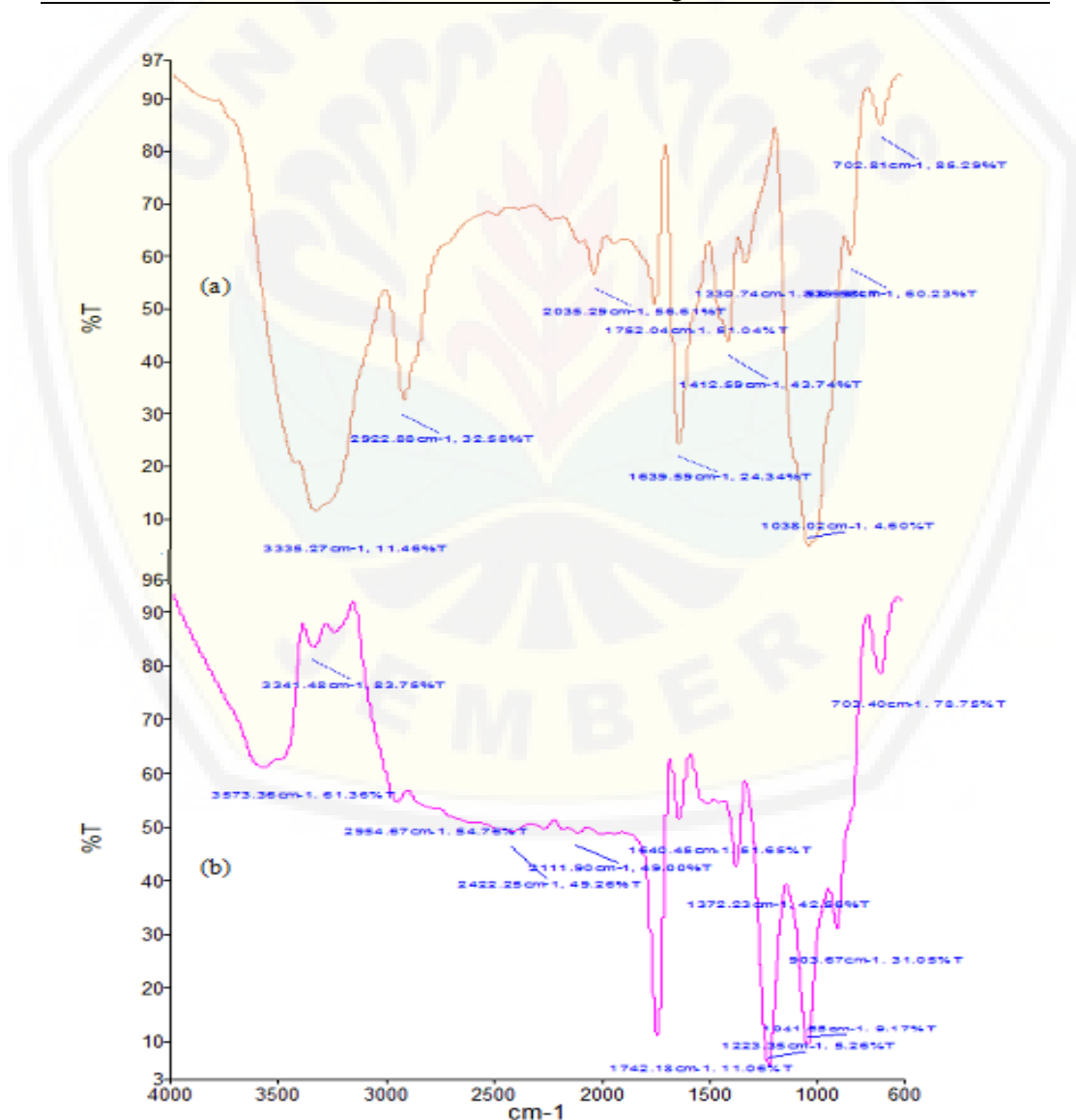


Figure 1 FTIR Spectra of (a) Bacterial Cellulose (b) Cellulose Acetate

Degree of substitution (DS) is a value that indicate average number of hydroxyl groups on glycosidic rings of cellulose substituted by acetyl groups [8]. DS value is affected on character and solubility of cellulose acetate. In this research, DS value and percentage of acetyl content were found at 2.62 and 41.413%, respectively. In comparison with commercial cellulose acetate (*Sigma Aldrich*), DS value and acetyl content were 2.17 and 36.943%. It was concluded acetylation process at 4 hours results cellulose diacetate. This result similar to previous research which variation of acetylation time (0.5 to 24 h), were found cellulose diacetate [15].

Thermal characteristic of cellulose acetate was examined by DSC. Figure 2 was DSC curve of commercial and synthetic of cellulose acetate. Thermal behaviour of synthetic cellulose acetate showed higher transition glass (T_g) and melting temperature (T_m) than commercial cellulose acetate (Table 2). It might cause by lower DS value and acetyl content of commercial cellulose acetate. However, commercial cellulose acetate had higher fusion enthalpi and crystallinity index. Both value were lower for synthetic cellulose acetate because of acetylation process had used high concentration of acid, hence, formation of crystalline region were influenced. Overall, there were no significant differences between both cellulose acetate.

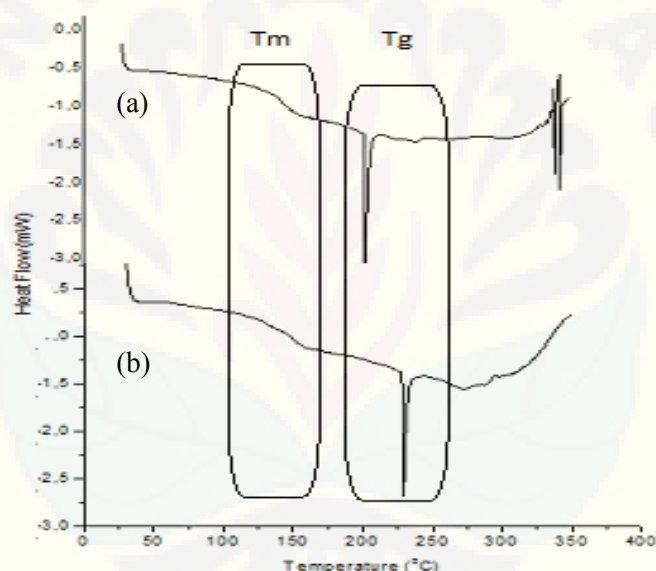


Figure 2 DSC curve of Cellulose Acetate

Table 2 Thermal Characteristic of commercial and synthetic cellulose acetate

Cellulose Acetate	T_g (°C)	T_m (°C)	ΔH_f (J/g)	X_c (%)
Commercial	134.5	203.5	23.68	40.27
Synthetic	150.2	228.8	15.43	26.24

3.2. Characterization of Blend Membrane PSF/CA

Blend membranes PSF/CA were prepared by phase inversion technique using water as non solvent agent. SEM images (Fig.3) of blend membranes PSF/CA showed porous structure for all variation of evaporation times. PEG-400 as additive in dope solution act as pore forming agent [6]. Evaporation times in phase inversion technique affected to the pore formation and membrane structure. Finger-like structure of pores were found on membranes at evaporation time 0 minutes (Fig 3a). Longer evaporation time induce shrinkages of the pore (Fig. 3b and 3c). It can be explained by kinetic aspect on membrane formation by phase inversion technique. The kinetic aspect was related to mass transfer during coagulation or solvent-non solvent exchange. Mass transfer was conducted by different concentration between solvent (NMP) and non solvent (water). When polymers solution had contact

with non solvent, liquid-liquid demixing was occurred. In the case of instaneous demixing, membrane pores were formed immediately induced porous membrane structure. On the other hand, delayed demixing was conducted membranes with more dense structure, due to slow pores formation [14]. Therefore, in the case effect of evaporation time, different porosity of PSF/CA membrane were obtained.

Porosity of PSF/CA membranes were decrease as increasing evaporation time (Fig.4). Membrane with no evaporation time had higher porosity by instaneous demixing process. However, membranes with 0.5 and 1 minutes evaporation time introduce to delayed demixing, hence, give lower porosity due to evaporation of solvent before immersion on water. When solvent had evaporated, concentration and viscosity of polymer solutions were increase, thus, solvent-non solvent exchange process was being restricted. Therefore, lower porosity were obtained for 0.5 and 1 minutes evaporation time.

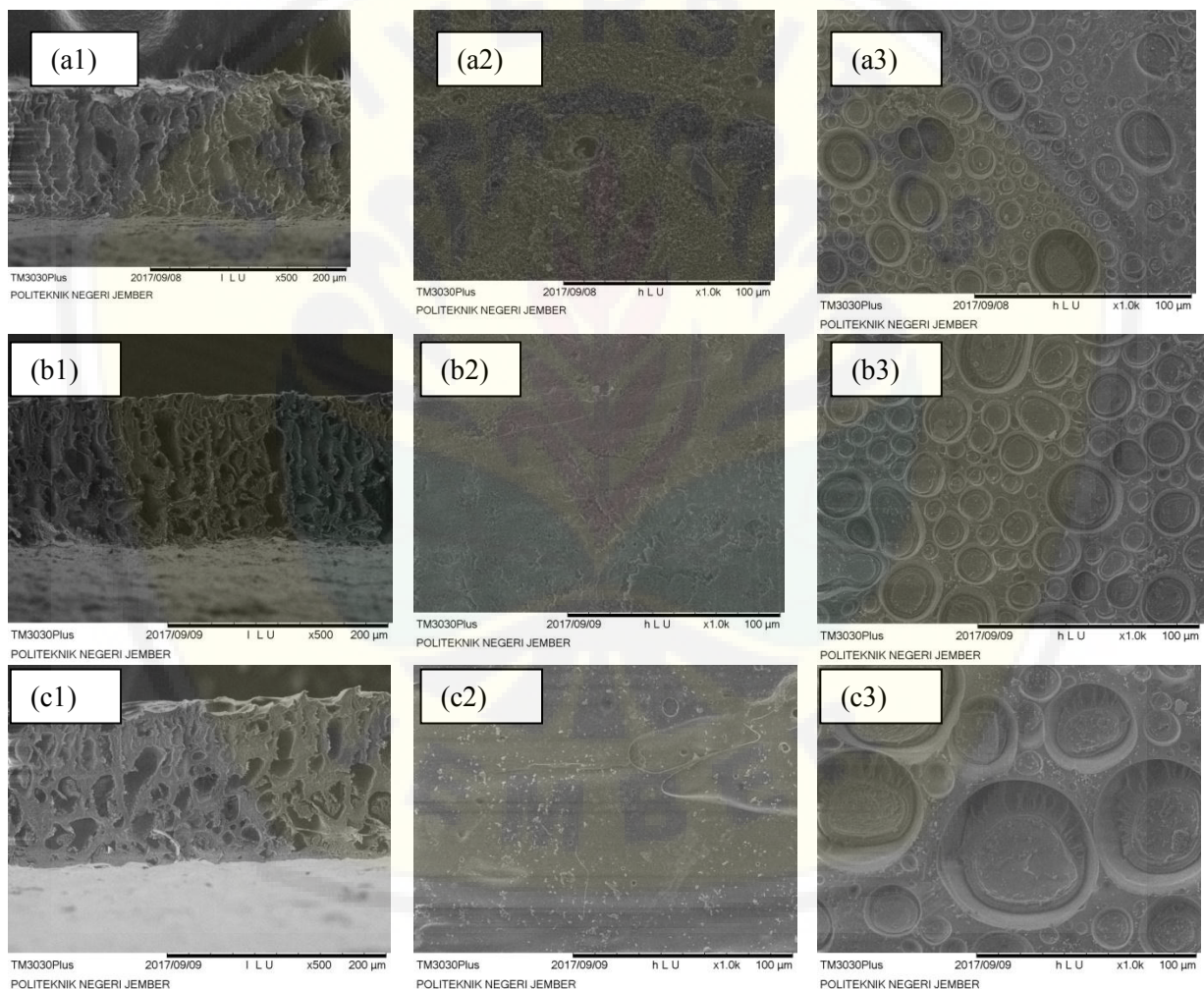


Figure 3 SEM Images of PSF/CA membranes on cross-sectional (1), surface (2) and bottom(3), respectively, at (a) no evaporation time (b) 0.5 min and (c) 1 min evaporation time

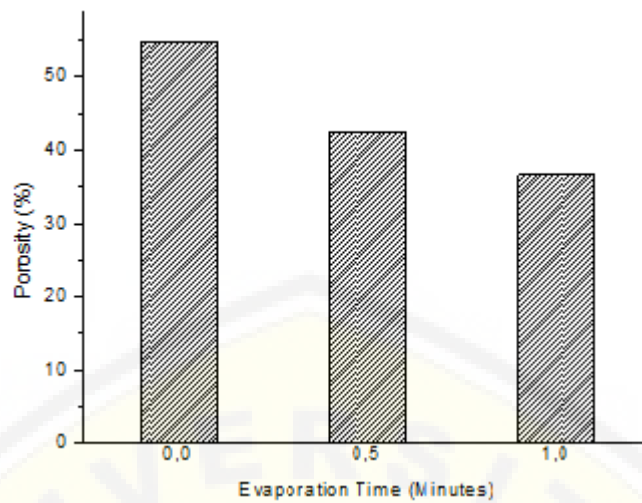


Figure 4 Porosity of PSF/CA membranes

3.3 Separation Performance of PSF/CA Blend Membranes

Pure water flux value of PSF/CA membranes with different coagulation time as shown on Fig.5. Adding small portion of CA on polymer solution had increase hydrophilic properties of the membranes. Increasing evaporation time has lowering water flux due to reduction of porosity. Moreover, delayed demixing (on 0.5 and 1 min) could produce denser pores structure, hence, reducing water flux. PEG-400 has play important role on pore formation. Pore network gave higher change for pure water run into the membranes produce higher water flux. Membrane PSF/CA without PEG has low water flux, about $9.8 \text{ L.m}^{-2}.\text{h}^{-1}$ [6]. It was 5-10 times lower than PSF/CA membranes with addition PEG-400.

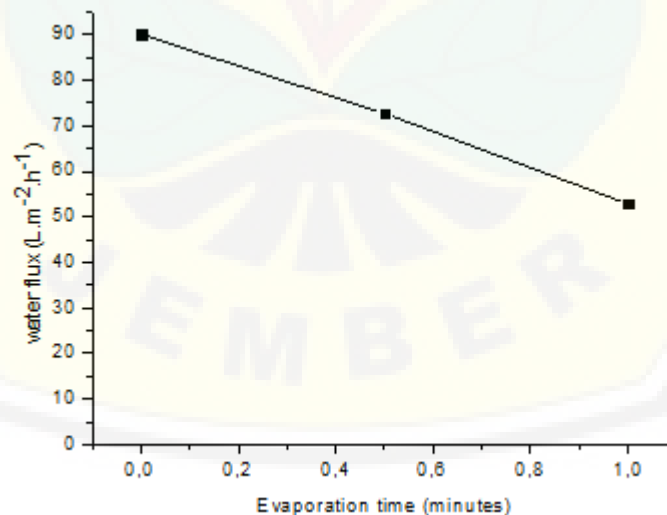


Figure 5 Water Flux on PSF/CA membranes

Separation performance of PSF/CA membranes were studied using surfactant stabilized dichloromethane/water emulsion. Water and permeate flux for membranes with different evaporation time depicted on Fig.6. Membrane with no evaporation time had higher flux and decrease for the next variation. Pores structure and porosity were play important role as describe in previous. The flux on membranes decrease sharply when water (J_{w1}) had replaced by permeates (J_p). It caused by fouling process had started on membrane and clog the pores. The flux remain increase for the second

filtration of water (J_{w2}) after cleaning process by water, indicate PSF/CA membranes had ability to recover the performance which determined by Flux Recovery Ratio (FRR) value.

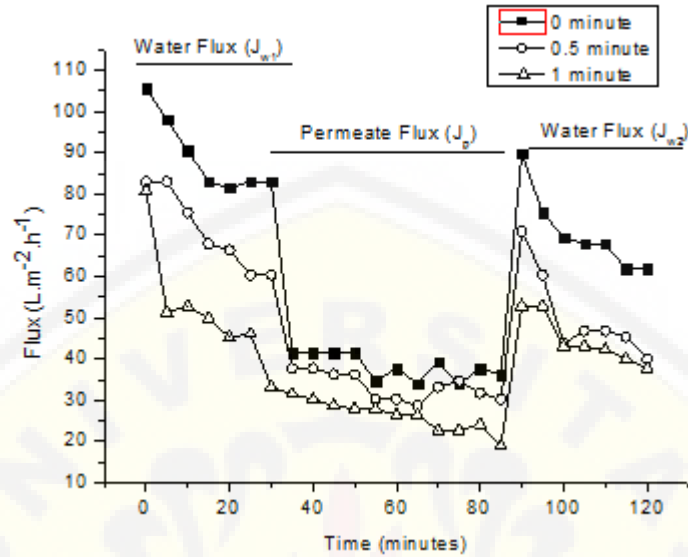


Figure 6 Water and permeate fluks on PSF/CA membranes

Flux recovery ratio (FRR) study show membranes with 0.5 minutes evaporation time had lower FRR (Fig.7). Fouling on membrane surfaces or pores has contribute to the recovery flux (J_{w2}). There were reversible (R_r) and irreversible (R_{ir}) fouling. Highest irreversible fouling was found on PSF/CA membrane with 0.5 minutes evaporation time (Fig.8), hence, decrease FRR value. Fig.8 was depict study of fouling resistance on PSF/CA membranes.

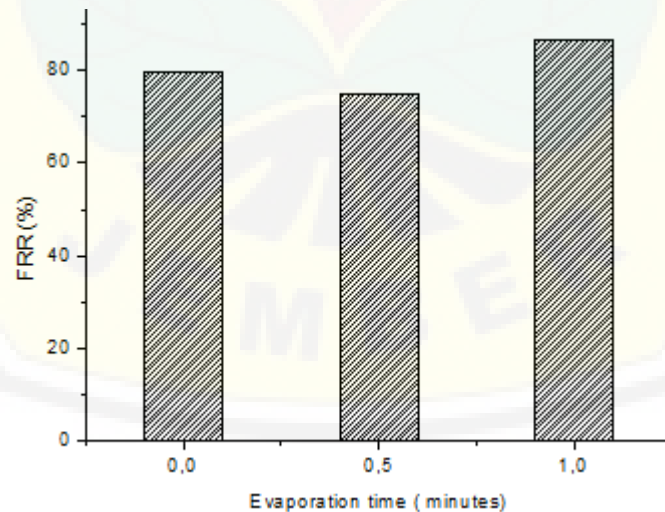


Figure 7 Flux Recovery Ratio (FRR) of Membranes PSF/CA

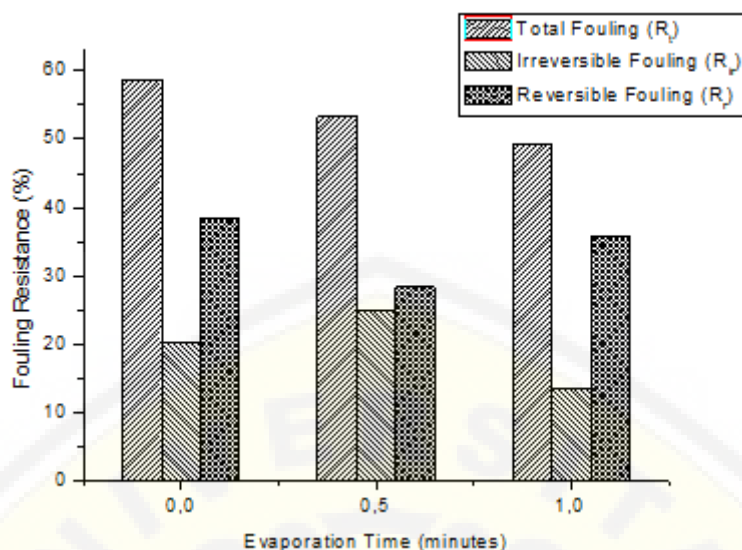


Figure 8 Fouling Resistance of PSF/CA membranes

Fouling resistance study on PSF/CA membranes show that increase on evaporation time had decrease the total fouling (R_t). The highest total fouling (R_t) was up to 58% for 0 minutes evaporation time. All of PSF/CA membranes show higher reversible fouling (R_r) that irreversible (R_{ir}) one. It might be caused by physical interaction between emulsion with membrane pores, therefore, it could be replaced from the pores by cleaning process with water. Irreversible fouling (R_{ir}) in this research did not exceed 25%, obtained by 0.5 minutes evaporation time, due to penetration and entrapping emulsion on membrane pores. As a results, blend membrane PSF/CA had good antifouling properties.

4. Conclusion

PSF/CA blend membranes were prepared with using bacterial cellulose as the source for CA. Characteristic of CA from bacterial cellulose were evaluated and show similar properties with commercial cellulose acetate (*Sigma Aldrich*). Evaporation time (0; 0.5; 1 min) before immersion were used as parameter to study characteristic and performance of PSF/CA blend membranes. SEM images show porous structure of membranes which influenced by presence of PEG-400. Porosity of PSF/CA membranes were decrease as increasing evaporation time. Membrane performance on filtration show highest flux obtained by 0 minutes evaporation time. FRR study show all membranes has recover the second flux up to 75%. However, membrane on 0.5 min evaporation time show lowest FRR due to irreversible fouling by entrapped emulsion on its pores. The highest total fouling (R_t) was 58%, but all of membranes show higher reversible fouling (R_r) than irreversible (R_{ir}) one. In conclusion, PSF/CA based on bacterial cellulose has good antifouling properties..

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