

Preparation and Characterization of Composite Cellulose Acetate/NaA Zeolite/Alumina Membrane and Its Application in Vetiver Oil's Pervaporation

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Abstract

Rice husk is an agricultural waste that contains cellulose. Rice husk's cellulose can be converted to cellulose acetate. Cellulose acetate (CA) is widely used as membrane's material for pervaporation. This material has high swelling degree that can influence its performance. The membrane's performance can be improved by addition of minerals such zeolite and alumina. The purpose of this study was to determine the characteristics and the best composition of CA/NaA zeolite/alumina composite membrane (CA/Na/Al) for pervaporation of Vetiver oil. Methods that has been done were acetylation of rice husk's cellulose, synthesis of composite membrane, characterization, and pervaporation of Vetiver oil. Focused characteristics of membrane were its swelling degree, mechanical strength, and its morphology. Composite CA/Na/Al membrane has lower swelling degree than CA/NaA membrane because the existence of alumina that fill CA matrix. The optimum mechanical strength was gained by adding 2% alumina to membrane. But, composite CA/Na/Al 3% membrane was the best composition due to its performance. Membrane performance including its flux and the percentage of vetiverol gained (C_p), with flux value $367.83 \text{ g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ and C_p value 15.58%.

Keywords: Rice husk, cellulose acetate, alumina, NaA zeolite.

Abstrak

Sekam padi merupakan limbah pertanian yang mengandung selulosa. Selulosa sekam dapat dikonversi menjadi selulosa asetat. Selulosa asetat (SA) dapat digunakan sebagai bahan baku membran pervaporasi. Membran SA memiliki derajat pengembangan (DP) yang tinggi sehingga menurunkan kinerjanya. Kinerja membran selulosa asetat dapat ditingkatkan dengan penambahan mineral berupa zeolit dan alumina. Penelitian ini bertujuan untuk mengetahui karakteristik dan komposisi terbaik membran komposit SA/zeolit NaA/alumina (SA/Na/Al) untuk pervaporasi minyak akar wangi. Metode yang dilakukan yaitu asetilasi selulosa sekam, sintesis membran komposit SA/Na/Al, karakterisasi membran, dan pervaporasi minyak akar wangi. Karakteristik membran yang diamati ialah nilai DP, kekuatan mekanik, dan morfologinya. Membran komposit SA/Na/Al memiliki nilai DP relatif lebih rendah dibanding membran komposit SA/NaA akibat adanya alumina yang membantu mengisi matriks SA. Membran memiliki kekuatan mekanik optimum pada penambahan alumina 2%. Namun, membran komposit SA/Na/Al 3% merupakan komposisi membran terbaik ditinjau dari kinerjanya. Kinerja membran meliputi fluks dan konsentrasi vetiverol yang diperoleh (C_p) dengan nilai fluks $367.83 \text{ g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ dan nilai C_p 15.58%.

Kata kunci : Sekam padi, selulosa asetat, alumina, zeolit NaA.

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1. INTRODUCTION

Rice husk is one of most agricultural waste that has not been used efficiently. Commonly, rice husk is used as firing fuel in

brick's production (Lintang and Soeswanto, 2011). Sukhla *et al.*, (2013) stated that rice husk consists of cellulose (35%), hemicellulose (25%), lignin (20%), and silica

(17%). Cellulose that contained in it can be purified and converted to cellulose acetate (CA) (Das *et al.*, 2014). CA commonly used as one of most membrane's material. CA membrane has been used as material of lithium ion's battery (Marfuatun and Pawestri, 2016), nanofiltration in water treatment (Kusworo *et al.*, 2011), and pervaporation of patchouli alcohol (Aisyah *et al.*, 2012). Even though, pervaporative CA membrane has high swelling degree because of its interaction with solvent or feed that can reduce its selectivity (Smitha *et al.*, 2004).

In order to decrease its swelling, membrane's density should be improved by blending process with minerals addition (Ernawati, 2014; Wang *et al.*, 2009). Minerals additive such NaA zeolite (Hamzah and Nasir, 2013) and metal oxide (Wang *et al.*, 2009) inside the membrane can result on its denser morphology. These minerals can fill free volume in CA matrix so the swelling can be reduced (Ernawati, 2014).

Dense membrane commonly used in separation process such gas permeation and pervaporation. Aisyah *et al.*, (2012) used dense CA membrane in pervaporation of patchouli alcohol from patchouli oil. This separation base on the difference polarity of its components. The purified component is patchouli alcohol which is the most polar from any other component in patchouli oil. Its alcohol content is the main parameter of its quality. This also happened in vetiver oil's component which vetiverol is the most polar component (Hanief *et al.*, 2013). Quality of vetiver oil can be determined by its vetiverol content (SNI 06- 2386-2006).

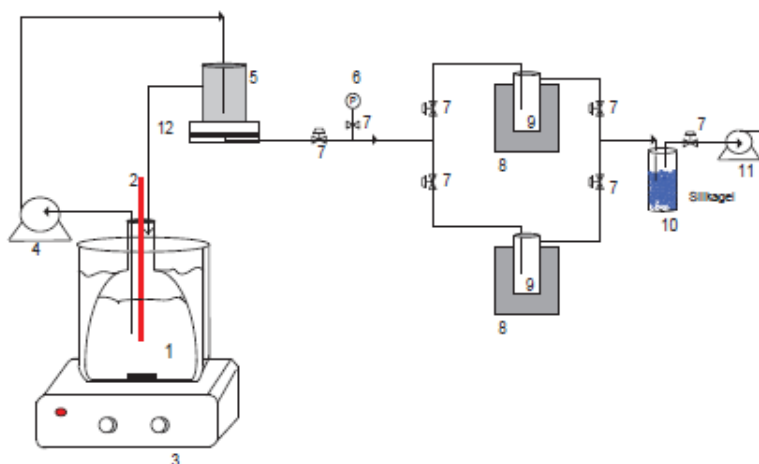
This research aimed to make and characterize composite CA/NaA zeolite/Alumina dense membrane, and to apply it in pervaporation of vetiver oil. The purpose of this research were to determine membranes characteristics and determine the best alumina composition based on membrane's characterization and its performance in pervaporation.

2. MATERIALS AND METHODS

Instruments and Materials

Apparatus used in this research were glass laboratory apparatus, buchner funnel, desiccator, oven, magnetic stirrer, glass plate, pervaporation apparatus, fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and universal testing machine (UTM).

Materials used in this research were : rice husk's cellulose from Inorganic-Physical Chemistry laboratory, Department of Chemistry, Padjadjaran University, vetiver oil from Samarang Village (Garut), aquadest (H₂O), alumina (Al₂O₃), acetic anhydride ((CH₃CO)₂O) (Merck), acetic acid glacial (CH₃COOH) (Merck), hydrochloric acid (HCl) (Merck), sulfuric acid (H₂SO₄) (Merck), oxalic acid (C₂H₂O₄) (Merck), acetone (C₃H₆O), ethanol (C₂H₅OH) (Merck), phenolftalein indicator (C₂₀H₁₄O₄) (Merck), potassium hydroxide (KOH) (Merck), methylene chloride (CH₂Cl₂) (Merck), sodium acetate anhydrate (CH₃COONa) (Merck), sodium hydroxide (NaOH) (Merck), sodium carbonate (Na₂CO₃) (Merck), sodium chloride (NaCl), and NaA zeolite (Wiko No.267-00595).



- Where :
1. Feed tank
 2. Thermometer
 3. Heater
 4. Circulation pump
 5. Pervaporation module
 6. Gauge apparatus
 7. spigot
 8. isoterm tube
 9. Cold trap
 10. Silica gel tube
 11. Vacuum pump
 12. Membrane

Figure 1. Pervaporation apparatus (Ernawati, 2014)

Cellulose Acetylation

Rice husk's cellulose (10 g) added into 250 mL acetic acid glacial and stirred by magnetic stirrer for 30 minutes. Then 1.6 mL of sulfuric acid and 97 mL acetic acid glacial were added to the mixture, then stirred for 25 minutes. Acetic anhydride (100 mL) added to the mixture and then stirred for 30 minutes, then the mixture stood for 14 hours at room temperature. The mixture then dropped onto stirred aquadest until it precipitated. Then the mixture was vacuum filtered to remove its solvent and water. The CA precipitate was washed with aquadest and dried in 70 °C for 24 hours (Kusumawati and Nurhayati, 2014).

Determination of Acetylation Degree

Acetylation degree of cellulose acetate was determined by placing 0.1 g CA inside erlenmeyer flask, then 5 mL sodium hydroxide 0.25 M and 5 mL ethanol were added. This mixture was left to stand for 24 hours. After that, 10 mL of hydrochloric acid 0.25 M was added to the system, which was left to stand for 30 minutes. Next, the mixture was titrated by standard solution of sodium hydroxide 0.25 M using phenolphthalein as indicator. Acetylation degree was determined by this equation (Filho *et al.*, 2008):

$$AD (\%) = \frac{[(Vb_i + Vb_t)\mu_b - V_a \cdot \mu_a]43.100}{m_{ca}}$$

Where:

AD = Acetylation degree,

Vb_i = volume of sodium hydroxide added to the system (mL),

Vb_t = volume of sodium hydroxide spent in titration (mL),

μ_b = concentration of sodium hydroxide (N),

V_a = volume of hydrochloric acid added to the system (mL),

μ_a = concentration of hydrochloric acid (N),

43 = molar weight of acetyl group (g/mol)

m_{ca} = weight of CA sample (g).

Preparation of CA/Na/Al Composite Membrane

CA powder 10% w/w was added to methylene chloride and stirred till homogeneous. Then, NaA zeolite and alumina were added to the mixture, which was stirred for 24 hours. Next, the mixture was placed in refrigerator for 24 hours. The mixture was

casted in glass plate. Membrane on the plate was placed inside the desiccator for 24 hours (Ernawati, 2014).

Determination of Membrane Swelling Degree

Membrane was cut with size 4 cm² then weighted. Then, membrane was placed immersed in vetiver oil at 40 °C for 24 hours. Next, membrane's surface was wiped till dry then weighted. The membrane's swelling degree was determined using this equation (Ernawati, 2014):

$$SD = \frac{Ma - Mb}{Mb} \times 100\%$$

where:

SD = swelling degree (%);

M = membrane mass before (b) and after (a) immersed

Membrane's Characterization

Membranes that have been produced where characterized by following characteristics : morphology, functional group, and mechanical strength using Scanning Electron Microscopy (SEM), Fourier Transform Infrared spectroscopy (FTIR), and Universal Testing Machine (UTM) instruments.

Pervaporation

Membrane was placed inside the module (Figure 1, point 5) horizontally in pervaporation apparatus. Then 100 grams of feed was placed inside feed tank (Figure 1, point 1) and it was heated at 40 °C. Next, Vacuum pressure (0.3 mbar) was added into the system, then permeate was taken every 1 hour for four times. Feed and permeate then were analyzed by titration method (SNI 06-2386-2006). Membrane's performance was determined by its flux using the following equation (Okumus *et al.*, 2003).

$$J = \frac{1}{A} \times \left(\frac{dm}{dt} \right)$$

where :

J = flux (g.m⁻².h⁻¹);

A = membrane surface area (m²);

t = time (h);

dm/dt = graph *slope* of permeate mass vs time

Determination of Vetiverol Content (SNI 06-2386-2006)

Determination of Ester Number Before Acetylation

A blank solution of 25 mL potassium hydroxide in ethanol 95% was refluxed for an hour. The solution was left to stand till cold, then titrated by 0.5 N hydrochloric acid. Sample test used the same procedure with placing 4 g \pm 0.05 g sample before refluxed. Ester number was calculated using the following equation:

$$E = \frac{56,1 (V_1 - V_0)N}{m}$$

Where:

E = ester number before acetylation,

56.1 = molecular mass of potassium hydroxide;

V_1 = volume of hydrochloric acid spent for blank solution's titration (mL),

V_0 = volume of hydrochloric acid spent of sample's titration (mL),

m = mass of sample (g),

N = concentration of hydrochloric acid (N).

Determination of Ester Number Before Acetylation

Vetiver oil (10 mL) added to 10 mL acetic anhydride and 2 grams of sodium acetate anhydrate then placed inside acetylation flask. The mixture was refluxed for 2 hours, then left to stand till cold. Next, the mixture was added by 50 mL aquadest, then heated at 40–50 °C for 15 minutes. The mixture was placed in room temperature. The mixture was moved to separation funnel and washed by 10 mL aquadest twice. The mixture was separated, then water layer was taken off. The oil layer was washed using 50 mL sodium chloride and 20 mL aquadest. Acetylated oil was shaken, then weighted. Next, 2 grams of oil was added by 2 mL of aquadest and 0.5 mL of phenolphthalein indicator. The mixture then added by 25 mL potassium hydroxide 0.5 N in ethanol. The mixture was refluxed for an hour, then fast cooled by adding 20 mL aquadest. The mixture was titrated by hydrochloric acid 0.5 N. Ester number after acetylation was calculated using following equation:

$$\text{Ester number} = \frac{28.05 (V_1 - V_0)}{m}$$

Where:

V_1 = volume of HCl spent on blank solution's titration (mL),

V_0 = volume of hydrochloric acid spent on sample's titration (mL),

m = mass of acetylated oil (g).

Next, content of vetiverol was calculated by this equation:

$$\text{Vetiverol content} = \frac{M (E_2 - E_1)}{561 - 0,42E_2}$$

Where:

M = molecular mass of vetiverol,

E_1 = ester number after acetylation,

E_2 = ester number before acetylation.

3. RESULTS AND DISCUSSION

Acetylated Cellulose Acetate

Rice husk's cellulose was converted to cellulose acetate (CA) through acetylation reaction within three steps. The first step, was *swelling*, cellulose fiber was swelled which made it has higher reactivity. In the second step, acetylation, sulfuric acid took role as catalyst which affected in speed up the reaction of hydroxyl group with acetic anhydride, and produced CA. In the third step, hydrolysis, CA was coagulated then a little amount of acetyl group of CA was hydrolyzed (Kusumawati and Nurhayati, 2014). The acetyl degree of produced CA was 31.05% and based on Gaol et al. (2013) it categorized as cellulose monoacetate.

Figure 2 showed absorbance peaks in FTIR spectra of cellulose and synthesized CA. Both spectra showed the vibration of hydroxyl group at wave number around 3400 cm^{-1} . But, in CA spectra, there was a transmittance depression because the intensity of hydroxyl groups was decreased, which has replaced by acetyl group. Next, CA spectra showed vibration of C=O bond of carbonyl ester group of acetyl around 1756 and 1754 cm^{-1} , which were not showed in cellulose spectra. In the wave number 1370 cm^{-1} showed the vibration of C-H bond from acetyl group in CA spectra. Then, in wave number around 1230 cm^{-1} showed the vibration of C-O group which connecting cellulose to acetyl group in CA spectra. Candido *et al.*, (2017) stated that the main characteristic of CA formation can be identified by its FTIR spectra, by following condition : a specific absorbance at 3400 cm^{-1} from hydroxyl group, at 1750 cm^{-1} from C=O carbonyl ester group, at 1360 cm^{-1} from C-H bond's vibration, and at 1220 cm^{-1} from C-O vibration which connecting cellulose to acetyl group. According to those absorbance peaks, could be stated that CA has been synthesized.

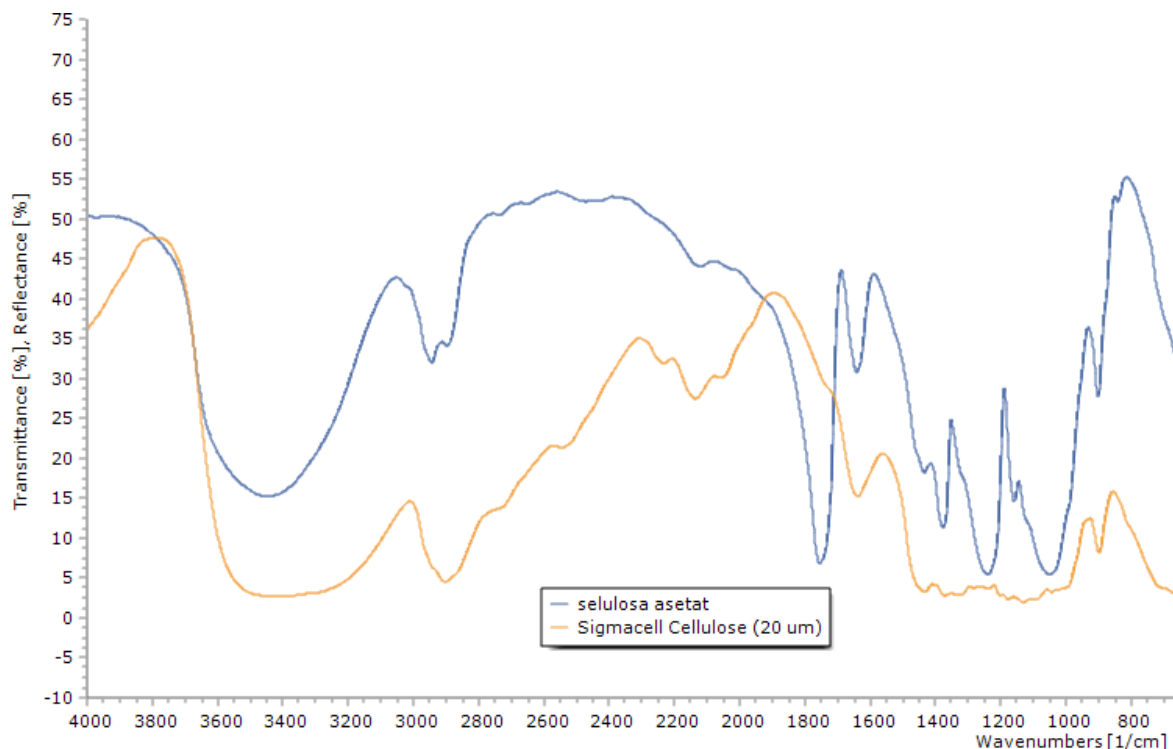


Figure 2. FTIR spectra of cellulose (orange) and synthesized CA (blue)

Comosite CA/Na/Al Membrane

Composition of composite CA/Na/Al membrane was CA 10% (w/w) with methylene chloride as solvent, NaA zeolite 5% of CA, and various concentration of alumina. Concentration of NaA zeolite used was 5% of CA mass, because Ernawati (2014) stated that in that concentration, membrane could show its best separation performance. Alumina was used in various concentration from 0, 1, 2, 3, and 4%. Wang et al (2009) have added alumina to CA membrane with various concentration from 0-4%, in separation of methanol/methylene-tert butyl eter (MTBE) mixture. The result was, CA membrane filled with 2% of alumina showed the best performances base on its flux and selectivity.

The stirring process could produce several bubbles that could be trapped inside membrane matrix. These bubbles could influenced membranes homogeneity which affected in void formation. This void could influenced membranes thickness and its mechanical strength. Therefore, debubbling process was done by placing the mixture in the refrigerator for 24 hours so the bubbles can be vanished and it could slow the evaporation of solvent (Ernawati, 2014). Membrane casting has been done by phase inversion technique,

by turning liquid phase of polymer to its solid phase in the controlled condition (room temperature) (Mulder, 1996).

Swelling Degree (SD)

Swelling degree of membranes showed in Table 1 and Figure 3. SD was one of most important parameters to determine dense membrane (Ernawati, 2014) and represented the interaction between solution and membrane's surface (Baker, 2004).

Table 1. swelling degree of CA, CA/NaA, and CA/Na/Al membrane

Membrane	SD / %
CA	9.66
CA/NaA	4.81
CA/Na/Al 1%	6.41
CA/Na/Al 2%	6.03
CA/Na/Al 3%	2.29
CA/Na/Al 4%	8.64

SD of CA membrane was 9.66%. This high SD could be happened from interaction of

solution to membrane, and then it trapped inside membrane because of membrane's free volume (Ernawati, 2014). Furthermore, membrane swelling could be happened from hydrogen bond between feed solution and hydroxyl group from CA matrix. The addition of 5% NaA zeolite showed to lowering SD from CA membrane, from 9.66 to 4.81%. It was assumed that membrane's free volume has been filled by zeolite (Ernawati, 2014). Alumina addition to membrane (composite CA/Na/Al membrane) with concentration 1% (w/w) raised its SD, this was assumed that the interaction of feed solution to membrane was increased, because the membrane was more hydrophile (Wang *et al.*, 2009).

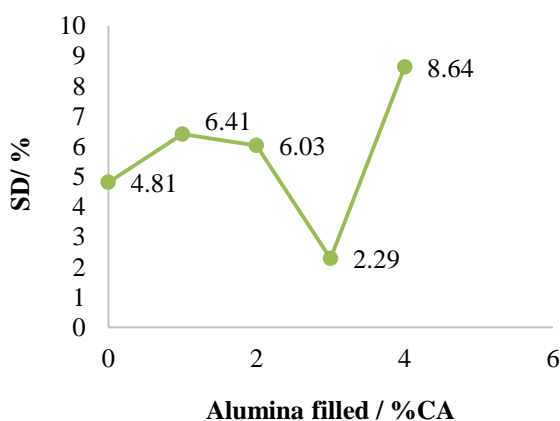


Figure 3. Effect of alumina filled to membrane's SD

CA/Na/Al 3% membrane has the minimum SD value, 2.29%. This decrease SD was assumed that membrane became denser resulting the less interaction between

membrane and feed solution. Alumina addition till 4% increase membrane's SD significantly. This was caused that overload of alumina could form a new porous layer on membrane's surface (Yang *et al.*, 2016) which increased its interaction with feed solution that affecting of rise of SD.

Membrane's Tensile-Strength

UTM was used to characterize membranes mechanical strength properties. The result was shown in Table 2. According to Table 2, CA membrane has mechanical strength 2.95 MPa. But, CA/Na/Al 2% membrane has the highest mechanical strength of all. This indicates that alumina particles (of each various concentration) interacted physically with CA/NaA membrane, resulting in improvement of its physical properties (Ahmad *et al.*, 2006). Interaction of alumina with CA/NaA membrane happened from adhesion force from both materials, besides, alumina filled CA matrix so the membranes became denser.

Addition of alumina more than 2% decreased its mechanical strength. This was assumed that optimum alumina addition to CA/NaA membrane was 2%. Next, alumina addition starting from 3% indicated the membrane was saturated of alumina addition. Based on its SD, alumina addition more than 3% could result in forming a porous alumina layer on membrane's surface which decreased its mechanical strength (Kiran *et al.*, 2018). Over amount alumina added to membrane made membrane more rigid than membrane with its optimum amount (2%).

Table 2. Membranes mechanical strength

Sample	Mechanical strength		
	F_{max} /N	$Stress_{max}$ /MPa	L_{max} /mm
CA	8.82	2.95	0.19
Ca/NaA	5.96	2.00	0.47
CA/Na/Al 1%	9.10	2.70	0.53
CA/Na/Al 2%	10.20	3.45	0.11
CA/Na/Al 3%	4.70	1.55	0.33
CA/Na/Al 4%	8.52	2.85	0.53

Membrane's Morphology

SEM micrograph of CA/NaA and CA/Na/Al membrane shown in Figure 4.

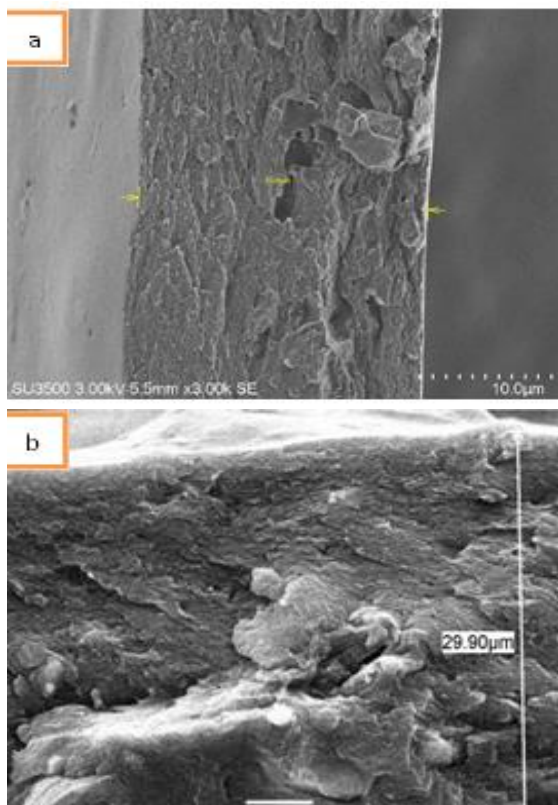


Figure 4. Cross section of CA/NaA (a) and CA/Na/Al 3% (b) membrane.

Composite CA/NaA membrane (a) has more homogeneous morphology than CA/Na/Al 3% membrane (b). Meanwhile, the morphology of cross section in CA/NaA membrane active side shown more porous than CA/Na/Al membrane. In this case, alumina filled CA/NaA matrix so membrane's porosity was decreased, in the other word, membrane became denser. The improvement of its density supported by its low SD value (2.29%) and almost half of SD value of CA/NaA membrane (4.81%).

CA/Na/Al Membrane's Application of Vetiver Oil Pervaporation

Based on membranes SD and mechanical strength, the lowest SD value and the highest mechanical strength were CA/Na/Al 2% and 3%. These membrane were compatible to pervaporation because of its denser morphology (proven by low SD value), high mechanical strength, have similar

properties to the target compound, which has polar properties (Ernawati, 2014). The performances of both membranes, with CA/NaA membrane as comparative, stated by its flux and vetiverol content (C_p) of permeate. Table 3 showed performance of each membrane.

Table 3. Membranes performance on pervaporation

Membrane	Flux / $\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$	C_p / %
CA/NaA	421.97	9.12
CA/Na/Al 2%	396.89	12.48
CA/Na/Al 3%	367.83	15.58

Based on SNI 06- 2386-2006, vetiverol content in vetiver oil was 2.4%. The C_p value in permeate of pervaporation using CA/NaA, CA/Na/Al 2%, and CA/Na/Al 3% membranes were 9.12%, 12.48%, and 15.58%, respectively. The improvement of C_p value showed that CA/Na/Al membrane is more selective in vetiver oil pervaporation. Along with the increasing of C_p value, membrane flux has decreased, with no significant influence. The flux decreased because of the denser membrane's surface, proven by the lower SD value than CA/NaA membrane (Table 2), with selectivity improvement. Flux of CA/NaA, CA/Na/Al 2%, and CA/Na/Al 3% membrane were 421.97; 396.89; and 367.83 $\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ respectively. Flux and selectivity were the main parameters in determining pervaporation membrane performance.

4. CONCLUSION

Composite membrane CA/Na/Al has denser morphology compared to CA/NaA membrane. Composite CA/Na/Al membrane with 2% alumina has the highest tensile-strength (10.2 N) while the lowest swelling degree owned by alumina loading of 3% (SD 2.29%). Performance of both CA/Na/Al 2% and 3% composite membrane in determined by its flux, respectively 396.89 and 367.83 $\text{g}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$, with C_p value 12.48 and 15.58 % respectively.

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