Preparation and characterization of cellulose acetate propionate membrane

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Article Info

Article history:

Received Apr 2, 2024
Revised Jul 15, 2024
Accepted Sep 3, 2024

Keywords:

Membranes
Cellulose acetate propionate
Phase inversion
Characterization
FTIR

ABSTRACT

Membrane technology is a technique in water and wastewater treatment that has many advantages. This study focuses on the manufacture and characterization of cellulose acetate propionate (CAP) membranes using the phase inversion method. The CAP is preferable since it is a biopolymer that is environmentally friendly and cheap. The production of CAP membrane was carried out using phase inversion method, and varied in 3 different concentrations of CAP i.e., 13, 14, and 15% wt., with additional material of polyethylene glycol (PEG). The characterization was conducted using scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) analysis to determine its morphology, pores, and functional groups. The results show that the membrane containing 13% wt. CAP exhibits higher porosity with more macropores than a membrane with 14 and 15% wt. CAP. However, membranes with higher concentrations of CAP show more uniform pores and fewer macropores. FTIR analysis confirmed the presence of functional groups in the membrane such as C-O, -CH_3, C=O, C-H, and –OH. It is also found that there is a shift in Wavenumber due to the increase in the concentration of CAP in each membrane.

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1. INTRODUCTION

A thin semipermeable layer that can isolate two levels by retaining certain components and transporting other components through the pores is defined as a membrane [1], [2]. The shape of the membrane depends on the physical properties of the layer such as porosity and water-base thickness, skeletal factors such as the weight of a substance passing through the layer, filtration time, nutrient concentration, and the various polymers used to make the layer. Most membranes are manufactured in the form of polymer composite membranes. Biopolymer-based membranes are widely developed such as cellulose acetate membranes. Cellulose acetate membranes are commonly used in reverse osmosis (RO), microfiltration (MF), and gas separation, and have high strength, high biocompatibility, high desalination capacity, high flow potential, and moderate stability [3]–[6]. These membranes are also widely used in various fields, such as plastics, coatings, medical, and optical film applications, as well as in various separation methods, e.g. filtration, dialysis, reverse osmosis, gas separation, and evaporation [7]–[10]. Compared to other separation processes, membrane technology has several advantages, including the use of membranes that do not change the structure or arrangement of the separated substances, can be operated at room temperature so that energy consumption is relatively low, and non-toxic because there are no other chemical additives, and is a clean technology because it does not create new waste [11]–[13]. Minimal equipment requirements are possible in membrane technology due to component mobility, and initial investment costs are lower than in conventional systems, no additional chemicals are required during operation, so no further waste is generated. This type of treatment is widely used in various applications, one of which is the removal and recovery of hazardous substances from textile wastewater [2], [14]–[17]. Membrane-based processes can be classified based on pressure differences, namely: microfiltration [18], ultrafiltration [19], and nanofiltration [20], [21]. The results of the separation are retentate and permeate. The mixture can be homogeneous or heterogeneous and can be solid, liquid, or gas [12]. Based on the material of manufacture, membranes can be divided into organic membranes and inorganic membranes [8], [12], [15], [22]–[24].

A method in membrane manufacturing in which a polymer is converted from a liquid phase to a solid phase by a specific control mechanism is called phase inversion [10], [15], [25], [26]. The phase inversion process occurs by evaporating the precipitating solvent, so that the evaporation can be controlled [27], [28]. The principle of phase inversion technology is based on the membrane production process which includes two phases, namely liquid phase and solid phase [15], [29]. The phase separation mechanism that occurs can be explained by a three-phase diagram with three main components, namely polymer, solvent, and non-solvent [30].

Cellulose acetate (CA) is one of the materials that has been most widely used as raw material for producing a membrane. The CA material has many advantages as membrane raw material such as being biodegradable, flexible, and good performance [6]. While cellulose acetate propionate (CAP) is another material that has similar characteristics to cellulose acetate but has a cheaper unit price than CA. The CAP material is a biopolymer with the chemical formula C76H14O49 [31] that has great potential to be applied in the manufacture of bioplastics. It has excellent biodegradability, transparency, and strength properties. The incorporation of propionyl groups gives CAP properties of water resistance, solubility, and compatibility with other polymers [13], [32]–[34]. Figure 1 presents the structure of CAP.

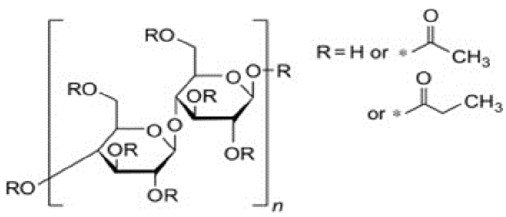


Figure 1. Structure of CAP [34]

CAP is one of the cellulose-derived polymers of organic acids, and the cellulose ester is of commercial importance [7]. CAP needs to be developed as a more useful material that exhibits enhanced properties for specialized applications while maintaining its environmental compatibility [12], [16]. It has an asymmetrical structure with a very thin active layer, can hold solutes in a rough buffer layer, is resistant to precipitation, and produces a balance of hydrophilic and hydrophobic properties are the advantages of CAP [3], [33], [35].

Various studies on membranes have been conducted with a variety of additives to various types of membrane base materials [33]. Optimization of the production method and production cost of a membrane has been widely studied [12], [36]. The CA material has many advantages as a membrane material; however, it has disadvantages in the unit price. While the CAP material has a lower price than the CA material, it has not been extensively utilized for membrane manufacturing. This research studies the manufacture of CAP biopolymer membranes and their characteristics at various concentrations with phase inversion techniques.

2. RESEARCH METHOD

2.1. Membrane fabrication

The materials used for membrane fabrication are CAP resin from Sigma-Aldrich, polyethylene glycol (PEG) 4000, acetone industrial grade as a solvent, and commercially distilled water. The manufacture begins with the preparation of a dope solution with concentration variations of 13 (by weight), 14, and 15% wt. of CAP and PEG 4000 with a concentration of 3% wt. The CAP and PEG were dissolved in acetone. The compositions of dope solutions for fabricating CAP membranes are shown in Table 1. The mixture of CAP-PEG-acetone was stirred at 200 rpm for 4 hours. After stirring for 4 hours, the solution was let to stand for 12 hours. The solution was then printed on a glass plate with the help of a printing knife with a thickness of 200 µm. The CAP membrane sheet was immersed in the coagulation bath containing distilled water for 5 minutes for the curing process. The membrane sheet was taken out from distilled water and then immersed in the 1% formaldehyde solution for 1 hour for preservation. After the preserving process was completed, the membrane was taken out and dried naturally at the ambient temperature and pressure, and ready for further analysis.

Table 1. CAP/PEG/Acetone composition for dope solutions

Composition			Target of membrane application	Membrane thickness (µm)
CAP (%wt.)	PEG 4000 (%wt.)	Acetone (%wt.)		
13	3	84	Microfiltration	200
14	3	83	Microfiltration	200
15	3	82	Microfiltration	200

2.2. Characterization of CAP membrane
2.2.1. Scanning electron microscopy (SEM) analysis

Characterization using SEM is to determine the pores and morphological structure of the CAP membrane. The cross-sectional observation data and pores on the membrane with a certain magnification are obtained by this SEM test [37]. SEM test was conducted using JSM-6510LA with a magnification range of 10-300,000, and resolution of 1-10 nm. This test was carried out at magnification of 3,000 and 10,000 times.

2.2.2. Fourier transform infrared (FTIR) analysis

The FTIR characterization test aims to determine the functional groups contained in the CAP membrane that has been fabricated. This test was carried out to see and confirm the presence or absence of CAP, acetone, and PEG contained in the CAP membrane. The test was carried out using a Shimadzu QATR10 single-reflection ATR accessory.

3. RESULTS AND DISCUSSION
3.1. Preparation of CAP membrane

The CAP membranes have been successfully prepared. The results of the preparation of the CAP membrane with the phase inversion method are shown in Figure 2. The summary of the physical appearance properties of membranes is shown in Table 2.

Figure 2(a) shows the physical appearance of the membrane with a CAP concentration of 13% wt., Figure 2(b) for the membrane with 14% wt. CAP concentration, and Figure 2(c) for the membrane with 15% wt. CAP concentration. Membranes with CAP concentrations of 13, 14, and 15% wt., show a similar physical appearance. No significant difference is observed in the appearance of the membrane surface. In general, the concentration of CAP is 13-15% wt. does not provide a difference in the physical appearance of the CAP membrane.

(a) (b) (c)

Figure 2. Printed CAP membranes with CAP concentrations of (a) 13% wt., (b) 14% wt., and (c) 15% wt

Table 2. Physical appearance characteristics of CAP membranes

CAP concentrations (%wt.)	Thickness (µm)	Color	Shape
13	200	Milky white	Polymer solids
14	200	Milky white	Polymer solids
15	200	Milky white	Polymer solids

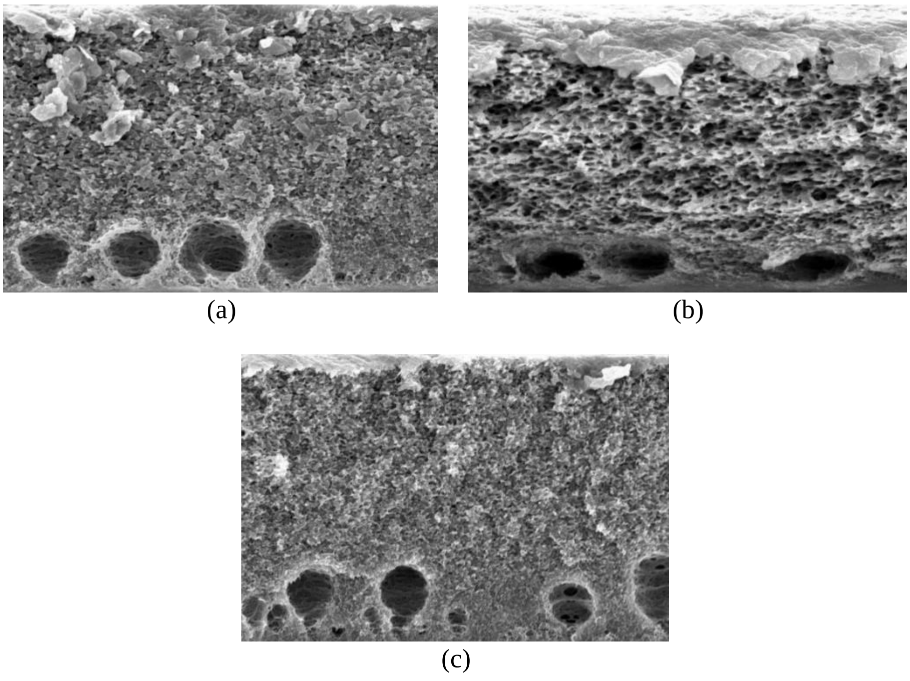
Table 2 shows that CAP membranes have similar physical appearance properties in the form of milky white polymer solids at different CAP concentrations. These results are similar to the results of a previous study [29] that uses two membrane raw materials, namely CA and polysulfone in the manufacturing process. The previous study also resulted in the membranes with a milky white color.

However, they have slight differences in surface roughness. The lower concentration of CAP gives a slightly rougher surface of the membrane. The differences in membrane roughness properties at various CAP compositions in the polymer blend solution can be explained by the membrane formation mechanism. The membrane in the coagulation bath undergoes a liquid-liquid de-mixing process influenced by the composition of CAP, PEG, and acetone in the polymer mixture solution. When the solution is introduced into the coagulation bath, it causes phase separation [38]. It has been also reported previously [39] that more macro-voids formed at lower concentrations of polymer in the manufacturing of polysulfone membranes. More macro-voids result in a rougher surface of the membrane.

3.2. Test result of SEM analysis

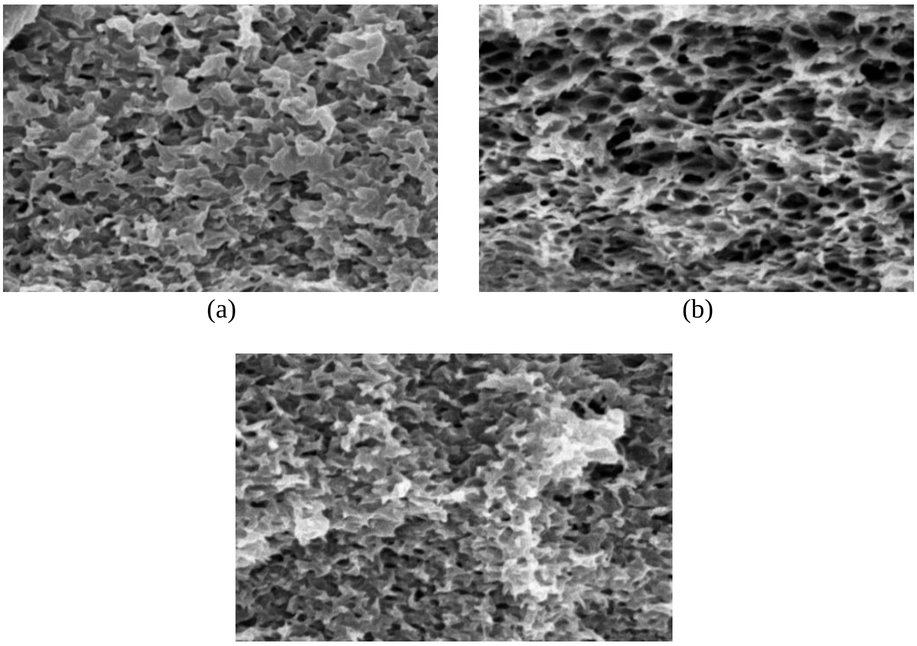
SEM analysis is useful to determine the morphology and pores of the CAP membrane [36], [40], [41]. Figure 3 shows the SEM test results of the CAP membrane at concentrations of 13% wt. (Figure 3(a)), 14% wt. (Figure 3(b)), and 15% wt. (Figure 3(c)), with a magnification of membrane pores of 3,000 times respectively. Figure 4 shows the SEM test results of the CAP membrane at concentrations of 13% wt. (Figure 4(a)), 14% wt. (Figure 4(b)), and 15% wt. (Figure 4(c)) with a magnification of 10,000 times respectively.

Figures 3 and 4 illustrate the SEM images of the CAP membrane which is an asymmetric membrane. It can also be seen that the pores in the membrane with a CAP concentration of 13% wt. are more numerous compared to the membrane with a concentration of 14 and 15% wt. CAP. It also indicates that the greater the concentration of CAP added to prepare the membrane, the smaller the pores obtained in the cross-section. Figures 3(c) and 4(c) show that highly porous structure with more dense pores. These results are similar to those of the previous research [30], which reveals the results of SEM analysis of the membrane morphology show that the greater the concentration of raw materials used in membrane manufacturing, the morphology and pores produced will be smaller. Meanwhile, according to the other research [42], the results of the SEM test on the cross-section show that the higher the concentration of additives used, the more pores are obtained in the membrane. However, a lower concentration of CAP results in a more macroporous structure in the membrane, while a higher concentration of CAP gives fewer macroporous and more microporous structure. The higher concentration of CAP also gives a more uniform macroporous structure of the CAP membrane.



(a) (b) (c)

Figure 3. SEM analysis of CAP membrane with concentration of (a) 13% wt., (b) 14% wt., and (c) 15% wt. at a magnification of 3,000 times



(a) (b) (c)

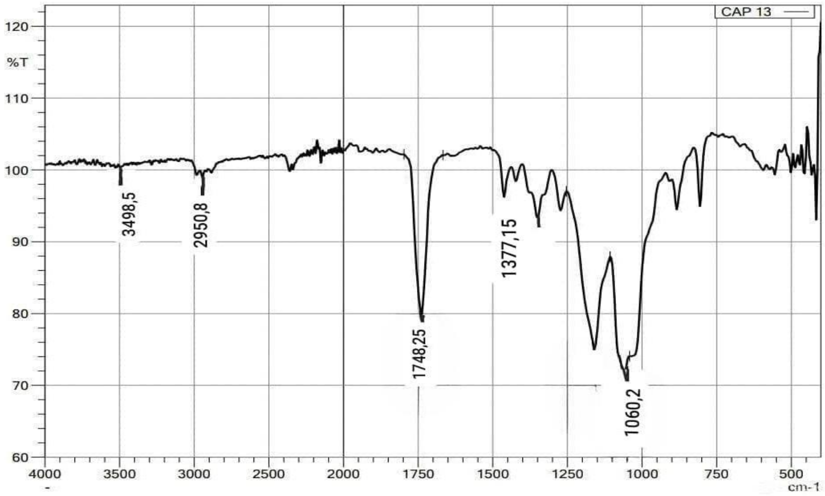
Figure 4. SEM analysis of CAP membrane with concentration of (a) 13% wt., (b) 14% wt., and (c) 15% wt. at a magnification of 10,000 times

3.3. Test result of FTIR Analysis

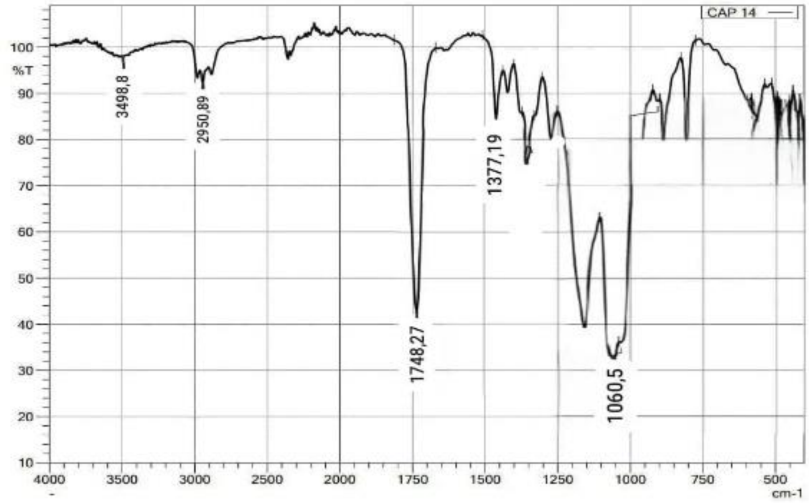
FTIR analysis on the membrane has the aim of knowing the presence of functional groups contained in the CAP membrane or identifying functional groups that exist in the CAP membrane [36], [41], [43], [44]. Figure 5 shows the FTIR spectra of CAP membranes for the CAP concentration of 13% wt. (Figure 5(a)), 14% wt. (Figure 5(b)), and 15% wt. (Figure 5(c)) respectively. The functional groups contained in the CAP membranes are summarized in Table 3.

The FTIR analysis is used to determine the presence or absence of PEG compounds (additives) and acetone solvents in the CAP membrane. If there are both compounds in the CAP membrane, there are new peaks in the results of FTIR analysis. The presence of new functional groups of the bonds between the CAP material with acetone solvent and additives (PEG 4000) or with one of the compounds will produce absorption peaks in different spectra.

Figures 5(a)-(c) show several functional groups that are detected, namely C-O, -CH3, C=O, C-H, and –OH in the CAP membrane. In the membrane with a CAP concentration of 13% wt. (Figure 5(a)), the functional groups were found at the peaks of 1060.2; 1377.15; 1748.25; 2950.8; and 3498.5 cm-1. While the FTIR results of membranes with a CAP concentration of 14% wt. (Figure 5(b)) and 15% wt. (Figure 5(c)), there are slight differences in the absorption peaks and their intensities compared to the membrane with a CAP concentration of 13% wt. (Figure 5(a)). However, they are still associated with the same functional groups These functional groups show that in the CAP membrane, there are not only CAP compounds but there are compounds or functional group bonds from PEG 4000 and acetone solvents. According to previous research [45] that explains the results of FTIR analysis on cellulose acetate butyrate membranes, there are C-H, C=O, and -OH functional groups. Where these functional groups indicate the presence of solvents, namely acetone solvent. While there is other research on CA membrane [42], reveals that there are several functional groups observed including: -OH; -CH3; -COOH; C-C; and -CH. The functional groups found in the membrane reported in the previous research [42], indicate the presence of raw material, solvent, and additive in the fabricated membrane. Therefore, the results of FTIR analysis in this study also show the presence of functional groups that are in accordance with the content of materials used i.e., CAP, PEG, and acetone.



(a)



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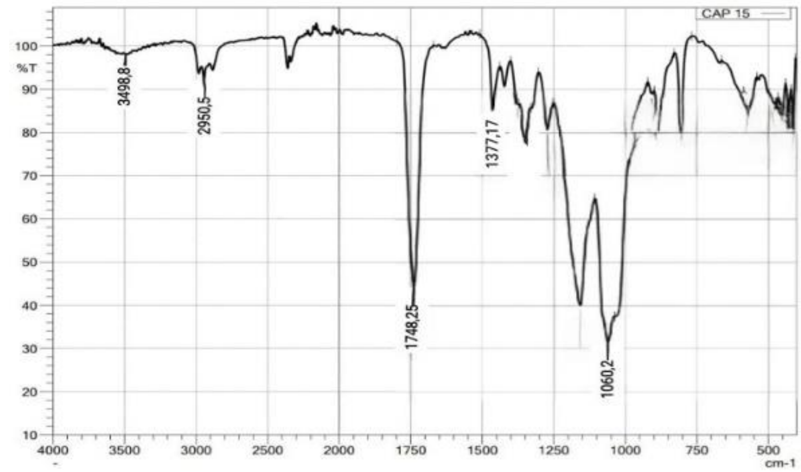

(c)

Figure 5. FTIR spectra of CAP membrane with CAP concentration of (a) 13% wt., (b) 14% wt., and (c) 15% wt.

Table 3. Functional groups of CAP membranes for various CAP concentrations from FTIR analysis

No.	Wavenumber (cm^{-1})			Functional Group
	CAP 13%wt.	CAP 14%wt.	CAP 15%wt.	
1	1060.20	1060.50	1060.70	C-O
2	1377.15	1377.17	1377.19	$-CH_3$
3	1748.25	1748.27	1748.29	C=O
4	2950.80	2950.89	2950.91	C-H
5	3498.50	3498.80	3499.01	-OH

4. CONCLUSION

The CAP membrane with PEG additive has been successfully fabricated using the phase inversion method. The concentration of CAP in the membrane was varied at 13, 14, and 15 %wt. The CAP membranes produced in this study have a milky white color in the form of a soft polymer. The results of SEM analysis show that the CAP membranes are asymmetric with pore structure and clarity that vary depending on the concentration of CAP. The CAP membranes with 13% wt. show a more uniform structure and clearer pores compared to those with 14 and 15% wt. FTIR analysis has revealed the presence of various functional groups, including C-O, -CH3, C=O, C-H, and –OH, with wave shifts observed concerning CAP concentration. These findings provide insight into the fabrication and characterization of CAP membranes for potential applications in microfiltration for water and wastewater treatment.

ACKNOWLEDGEMENTS

The authors would like to thank Universitas Ahmad Dahlan, Yogyakarta, Indonesia for providing financial support through research funding with contract number: PD-250/SP3/LPPM-UAD/VIII/2023.

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