The Influence of Casting Machine Speed in Cellulose Acetate Membrane Preparation

Luqman Buchori
Department of Chemical Engineering, Faculty of Engineering, University of Diponegoro Semarang
Jl. Prof. Sudarto, SH No. 1 Tembalang, Semarang, Phone 062-24-7460058

luqman.buchori@gmail.com

Abstract **- Membranes are being increasingly used as a valuable separation tool in laboratory as well as in industrial processes. Continual development of new membrane materials is crucial to sustain and expand the growing interest in this technology and modern polymer chemistry is highly proficient in tailoring polymers with desired properties such as increased mechanical, thermal and chemical stability. Cellulose acetate (CA) is one of the membrane polymers that has been used for aqueous based separation and used as both reverses osmosis (RO) and ultrafiltration (UF) membranes. This paper presents investigation of influence of casting machine speed in membrane preparation. CA is used in this study. That was CA-398-30 with average acetyl content 39.8 wt %. Non solvent used is water and the solvent is 99.7 % organic Dimethyl Sulfoxide (DMSO). The homogenous CA/DMSO solutions were prepared by dissolving CA in DMSO solvent. The CA concentration were varied 13 and 16 % while casting solution speed were varied from 20 to 80 mm/sec with distance cutting machine were fixed at 14 cm. In addition, non solvent in coagulation bath also were varied by 100 % water and 10 % DMSO in water. The results of this experiment describe effect of casting machine speed to water permeability. The effect of CA concentration to water permeability shows the adversative result between 13 and 16 % CA in 100 non solvent water. The similar result also observed in 10 % DMSO in water.**

Keywords : Cellulose acetate membrane, casting machine speed, membrane separation

I. INTRODUCTION

Membranes are being increasingly used as a valuable separation tool in laboratory as well as in industrial processes. Continual development of new membrane materials is crucial to sustain and expand the growing interest in this technology and modern polymer chemistry is highly proficient in tailoring polymers with desired properties such as increased mechanical, thermal and chemical stability [1-3].

The development of new polymer material is one of the important elements in the quest for advances and improvement in membrane Technology. The various membrane processes and range of particles diffusing through or retained are based on the membrane pore sizes. Membrane processes like microfiltration (MF), ultrafiltration (UF), reverse osmosis (RO), electro dialysis (ED) etc., are used individually or integrated appropriately with other traditional techniques, in many industries for the recycling of rare metals, toxic chemicals, polymer binders etc., Cellulose acetate (CA) was one of the first membrane polymer that has been used for aqueous based separations and used as both reverse osmosis (RO) and Ultrafiltration (UF) membranes[4-6]. So, how to prepare CA membrane or what is the optimum condition of CA membrane preparation are still become the main issue in membrane preparation. This research studied the influence of casting machine speed (which equivalent to exposure time of casting solution before immersed in coagulation bath) to morphological of membrane.

II. MATEIALS AND METHOD

A. Materials

Cellulose acetate used in this study was CA-398-30 with averaged acetyl content 39.8 wt% supplied by Eastman Chemical Company. Non solvent water used from tap water obtained from laboratory, while the solvent 99.7 % organic Dimethyl Sulfoxide (DMSO) were purchased from Merck.

B. Polymer solution preparation

The homogenous CA/DMSO solutions were prepared by dissolving CA in DMSO solvent by stirring for 12 hours until complete dissolution obtained. The CA concentration were varied 13 and 16 % while casting solution speed were varied from 20 to 80 mm/sec with distance cutting machine were fixed at 14 cm. In addition, non solvent in coagulation bath also were varied by 100 % water and 10 % DMSO in water. Composition on casting solution and non solvent in coagulation bath as be shown in Table 1.

TABLE I.
CASTING SOLUTIONS COMPOSITIONS AND FILM CASTING CONDITIONS

CA concentration, % in DMSO	Coagulation bath	Membrane/Casting machine speed (mm/sec)			
		CA1	CA2	CA3	CA4
13	100 % water	20	40	60	80
16	100 % water	20	40	60	80
13	10 % DMSO in water	20	-	-	80
16	10 % DMSO in water	20	-	-	80

C. Membrane preparation

Polymer solutions were cast on a glass plate with a thickness of 200 μ m, then immediately immersed into a water bath for 45 minutes. The thickness of the casting solution was adjusted with a casting knife. Before immersing to water bath, casting solution speed were varied to allow evaporation at room temperature and humidity. Speed of glass plate casting solution were set and moved automatically by computer. The resulting membranes were kept in the water bath for a day to remove the residual solvents. The casting machine angle were fixed at 30^{o}.

D. Characterization of membranes

All the membranes were characterized in terms of their pure water flux, morphological studies and so on as follows.

Water Permeability (WP). The experiments for pure water permeability (WP) and the retention of the membranes were carried out by Amicon stirred cell in dead end mode. Before characterization, all membranes were compacted through permeation experiments of deionized water at a pressure of 2 bar and for a period of 0.5 h. This avoids pressure effects on membrane structure in subsequent experiments. Stirred cell has effective surface area 0.000314 m^2. The transmembrane pressure was 0.5 bar. Milli-Q ultrapure water was used to determine the pure water permeability. From the measured values, the WP was determined from the expression,

$$WP = \frac{Q}{A.\Delta T.\Delta P}$$

where WP water permeability (L/m2.h.bar), Q the quantity of water permeated (L), ΔT the sampling time (h), A the membrane area (m^2), and ΔP *transmembrane pressure (bar)*

Morphological study. The top surface and cross-sectional morphology of the membranes were studied using scanning electron microscopy(SEM). Membranes samples were freeze-fractured using liquid nitrogen and coated with a thin layer of gold using an SPI-Module sputter. The cross-section and membrane surface of the flat-sheet membranes was examined using scanning electron microscope.

III. Results And Discussion

A. The effect of the casting machine speed time to membrane morphology

In this study, the effects on the membrane performance mainly came from the factor including the exposure time of phase separation induced by water vapor. The exposure time of casting solution before contact to non solvent in the coagulation bath were varied by varying casting machine speed. The casting machine speed of 20, 40, 60, and 80 mm/sec will correspond to 7.00, 3.50, 2.33, and 1.75 sec exposure time since the distance of cutting machine to coagulation bath were fixed at 14 cm. Fig. 1 shows the relationship between the membrane morphology and the exposure time of phase inversion induced by water vapor.

Fig.1 shows that the cross section morphology of all membrane produced have asymmetric structure as in general case of membrane produced by inversion phase [7-9]. All membranes have thin layer (skin layer) and porous layer (support layer). However, all membrane produced not clear exhibit the pore of void in the support layer as well as skin layer. This is also shown in the Fig. 2.

B. The effect of the casting machine speed to water permeability

The effect of the casting machine speed to water permeability were studied by varying the casting machine speed, then the membrane produced were measured the flux water permeability. The experiment observed completely shown in Fig. 3.

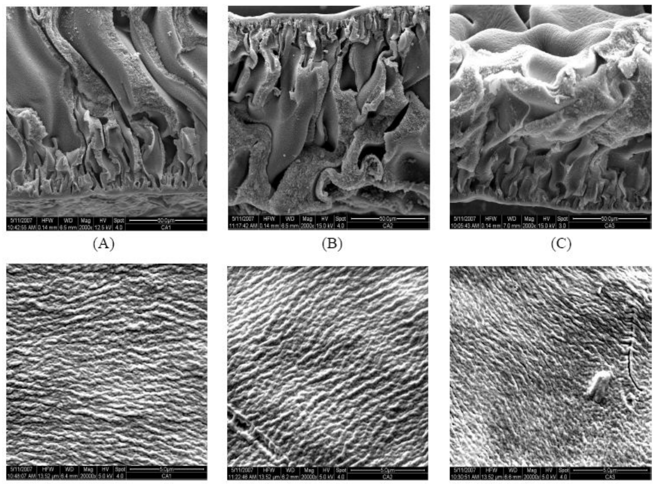


Fig. 1. SEM images (2000 x) of membrane surface morphology (below) and cross section morphology (above) made of 13 % CA in DMSO with different exposure time : (A) 1.75 sec (B) 2.33 sec; (C) 7.00 sec

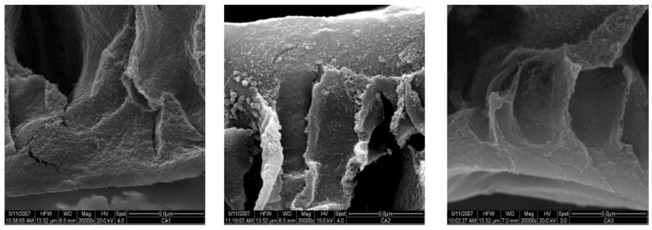


Fig. 2. SEM images (20.000 x) of membrane cross section morphology made of 13 % CA in DMSO with different exposure time : (A) 1.75 sec (B) 2.33 sec; (C) 7.00 sec

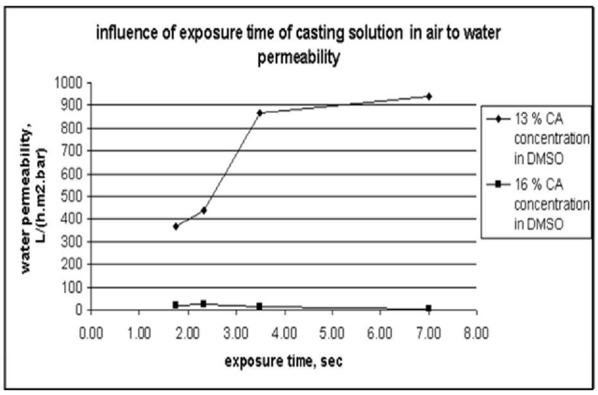
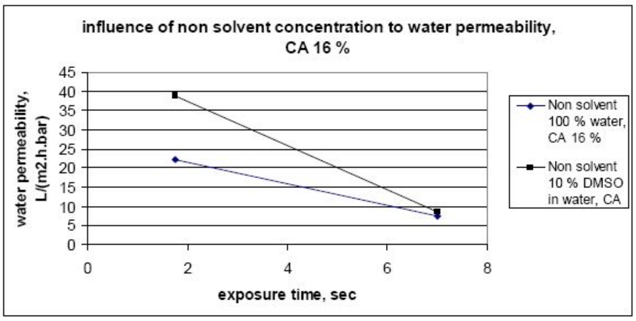


Fig. 3 The influence of the casting machine speed to water permeability

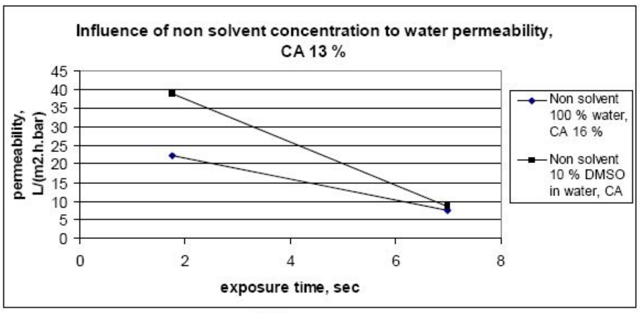
Fig. 3 above shows that CA membrane produced from 13 % CA in DMSO exhibit different trend in water permeability in compare to CA membrane produced from 16 % CA in DMSO. With the membrane produced from 13 % CA in DMSO, the increasing of exposure time also will increase the water permeability. This is different with membrane produced from 16 % CA in DMSO which is the water permeability will tend to decrease if the exposure time decreased. In general, the higher exposure time will decrease pore size as a consequence that membrane resulted tend to make dense layer. The complex mechanism may be involved in membrane formation as the consequence of the increase CA concentration e.g viscosity and density of solution during phase change in membrane preparation. The further researcher may continue in the next research what is the mechanism involved.

C. The influence of the non solvent concentration to water permeability

The non solvent used varied from 0 and 10 % DMSO in water. The experiment observed completely shown in Fig. 4.



(A)



(B)

Fig. 4 The influence of the non solvent concentration to water permeability

Fig. 4 shows that the 10 % DMSO in non solvent water will increase the water permeability of membrane produced. The similar trend occurred in both two kind of membrane that are 13 % CA and 16 % CA in DMSO. In addition, both two kind of membrane also exhibit the similar trend in the influence of exposure time to water permeability, the increase of the exposure time will tend to decrease the water permeability.

IV. Conclusion

The cross section morphology of all membrane produced have asymmetric structure as in general case of membrane produced by inversion phase. The membrane produced consist of the support and skin layer. All membranes have thin layer (skin layer) and porous layer (support layer). CA membrane produced from 13 % CA in DMSO exhibit different trend in water permeability in compare to CA membrane produced from 16 % CA in DMSO. The 10 % DMSO in non solvent water will increase the water permeability of membrane produced. The similar trend occurred in both two kind of membrane that is 13 % CA and 16 % CA in DMSO. In addition, both two kind of membrane also exhibit the similar trend in the influence of exposure time to water permeability, the increase of the exposure time will tend to decrease the water permeability.

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