PROCEEDING

INTERNATIONAL SEMINAR ON ENVIRONMENTAL SCIENCE

"Environmental based on Science Research for a Better Life

Gedung Seminar E University of Andalas Padang, Indonesia

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Andalas University

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PREFACE
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Assalammu'alaikum Wr. Wb

All praises be to Allah, the Almighty, the creator of the world. May His peace and blessing be upon our prophet Muhammad SWT, the leader of messengers and guide of faithful.

Environmental problems are now becoming the hot issues which have need to solve by all of us, because these issues directly effect the quality of humans life on earth. Therefore, as scientist we have to give more effort to give solutions on those issues.

This proceeding is useful as a guide to fix those issues, as the first step to keep the environmental safe for our next generation.

Padang, 2012
Vice Rector III
Andalas University

Prof. Dr. H. Novesar Jamarun, MS
NIP. 1962 0506 198811 1001

PREFACE
DEAN OF THE FACULTY MATHEMATICS AND NATURE SCIENCE
ANDALAS UNIVERSITY

Assalamualikum, wr.wb,
All praises be to Allah, the Almighty, the creator of the world. May His peace and blessing be upon our prophet Muhammad SWT, the leader of messengers and guide of faithful.

I would like to thank all of you who participated in this International seminar which is held here in Padang, and specially, key note speakers that came here, and organizing committee with hard work to prepare this seminar. For the students, I think, this event is learning process that to be important in the future time.

I am proud that so many experts in the field of this seminar decided to take part in the event that has been co-organized by our chemistry students.

I am confident that the seminar will provide a great opportunity for an international and national gathering for sciences and technology from different nation.

As we know, at the moment environmental and energy are great problem for us. In this case, research is one of that we can do to solve these problem and important role to improve the quality of life. I do hope that, this seminar provided platform for scientist and technologist to meet and share ideas, achievement as well as experiences through the presentation of papers and discussion.
Wassalamualikum ww

Padang, 2012
Dean of the Faculty
Mathematics and Nature Science
Andalas University

Prof. Dr. H. Emriadi, MS
NIP. 196204091987031003

FOREWORD
MAJOR HEAD OF CHEMISTRY
UNIVERSITY ANDALAS

Assalanmmu'alaikum Wr. Wb
Thanks be to God praise for all the abundance of His grace and guidance. Salawat coincided greet Rasurullah SAW.

International Seminar on the theme "Environmental Based on Science Research for a Better Life". This is proof that we as chemistry scientist aware that environmental problems are exist, because the environment is one of subarea in which we understood better as a chemistry scientist.

The environmental issues now rise up with the rapid of science and technology development that is not coupled with environmental awareness. The waste that is not handled properly, carelessly and discarded on the right spot, turn raises into new porobloms for the environment, also to the people who were around the neighborhood.

Therefore, the presence of this International Seminar, and the summarize of various studies to address various environmental problems that have been packaged in this procedding can provide solutions and contribute little to the existing problem. Hopefully it can be optimized and maximized its use.

Padang, 2012
Chairman of the Chemistry Department
Andalas University

Dr. Adlis Santoni
NIP. 1001 1962 0506 198811

Content

Study of Fourier Transform Infrared Spectroscopy and Scanning Electron Microscope of Cellulose Acetate Membrane from Wastewater of Tapioca.

Betty Marita Soebrata, Sri Mulijani, Ismiaini Nurpatria Putri

Department of Chemistry, Faculty of Mathematics and Natural Sciences,
Bogor Agricultural University
Email: bettymarita@gmail.com

ABSTRACT. Wastewater of tapioca is waste product from starch precipitation or separation process from its water in making tapioca. This wastewater still contains carbohydrates such as glucose, so it can be used as raw material for making cellulose and cellulose acetate (CA) membrane. *Nata de cassava* was made by fermentation process using *Acetobacter xylinum* in wastewater of tapioca as the fermentation media. Then, the *nata* was dried and grinded to produce bacterial cellulose (BC) powder. BC powder was reacted with acetic anhydride to produce CA powder. CA powder was dissolved to acetone solvent (appropriate with an acetyl degree of CA) with 16% w/v composition and then formed to be a membrane. These CA membrane was characterized with Fourier Transform Infrared (FTIR) and Scanning Electron Microscope (SEM). Analysis from FTIR spectrum showed that CA was formed, by the existence of absorption band of C carbonyl at 1725.92 cm-1 wavenumber and C-O acetyl at 1061.37 cm-1. The result of SEM analysis showed that the membrane which has been produced was the microfiltration and asymmetric membrane with the diameter pore approximately of 0.37-0.95 µm. CA powder that has been produced had a water content of 21.39% and an acetyl degree of 40.38% (equal with substitution degree of 2.51).

Keywords: cellulose, cellulose acetate, nata, bacterial cellulose, membrane, , FTIR, SEM

1. Introduction

Cassava *(Manihot esculenta)* is a clump of food crops from the Americas and has another name *ubi kayu* or *singkong*. In Indonesia, cassava is an important source of carbohydrates after rice, with a carbohydrate content of 34.7% (Soetanto 2001). Cassava can grow even in infertile soil, in the tropical climate. Indonesia is currently the third largest cassava producing countries in the world after Brazil and Thailand because many Indonesian people have switched from planting rice to cassava farming (Sumiyati 2009). According to Badan Pusat Statistik (BPS 2011), the amount of cassava production in Indonesia in 2009 was 22,039,145 tons and in 2010 increased to 23,908,459 tons (preliminary figures).

Cassava can be consumed directly as food or used as a raw material of food industry, one of which is the tapioca industry. The process of making the tapioca in the industry will generate solid waste (*onggok*) and liquid waste. Tapioca liquid waste can be generated from the washing stage of raw materials (cassava) and also the deposition process to separate the starch from the water. The process of making tapioca in large industries and small industries in general includes the stages of skin peeling, washing, scrapping, extraction, starch deposition, and drying.

Wastewater from tapioca industry which is still new is creamy color, while the color of spoiled waste is dark gray. Turbidity that occurs on the waste caused by the presence of organic materials, such as soluble starch, microorganisms, and other colloid materials. Tapioca liquid waste from the precipitation has a BOD value of 1450.8-3030.3 mg L^{-1} with an average of 2313.54 mg L^{-1}, COD of 3200 mg L^{-1}, dissolved solids 638-2836 mg L^{-1}, and the amount of cyanide (CN) of 19.58-33.75 mg L^{-1} (Vegantara 2009). Therefore, if the waste is discharged into the environment that will cause environmental pollution, such as bad smell and some wells are not suitable for consumption.

The current technology has not been able to separate the starch which dissolved in water so that liquid waste which is released into the environment still contains starch. The liquid waste will decompose naturally in water bodies and cause a bad odor. The smell is generated in the process of decomposition of compounds containing nitrogen, sulfur, and phosphorus of the protein material (Olive 1999 and Hanifa *et al.* 1999). Another alternative that can be done to treat this wastewater into products that can be exploited further is to process them into so-called *nata de cassava.*

Nata formation requires the element C that comes from glucose and N and O elements derived from the addition of ammonium sulfate. These elements are used as nutrients and carbon sources for bacteria *Acetobacter xylinum* in order to form a nata. Tapioca wastewater containing glucose as the carbon element can therefore be used as a good medium to stimulate the growth of bacteria such as forming nata. Therefore, tapioca liquid waste could potentially produce nata. Nata in the form of cellulose produced by bacteria that can be referred to as bacterial cellulose (BC). In addition, nata can be characterized as a composite of carbon fibers into a solid form and porous so nata potential to be utilized as a cellulose membrane. However, the cellulose membrane did not have a high commercial value. Therefore, further processing is needed to turn it into a membrane of cellulose acetate (CA) which is more valuable than cellulose membrane.

Cellulose triacetate (primary CA) obtained from the reaction of cellulose with acetic anhydride and H_2SO_4 as a catalyst. Acetylation is an important stage in the manufacture of CA as it relates to its ability to dissolve in certain organic solvents in the process of membrane manufacture. CA solubility in organic solvents is influenced by the levels of acetyl (Desiyarni 2006). Setting levels is carried out by reaction of acetyl hydrolysis using dilute acetic acid to obtain the appropriate degree of substitution.

This study aims to make membrane-based CA tapioca liquid waste, and identify it by Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscope (SEM). The analysis was performed to characterize the functional groups and types of CA membrane.

2. Materials and Methods

Preparation of *Nata de Cassava*

Tapioca liquid waste that has been filtered is taken in increments of 1 L was then added sugar as much as 10% (w/v) and ammonium sulfate as much as 0.5% (w / v), the mixture is heated to boiling, stirring frequently, then when it has been boiled it is poured into the fermentation container which has been prepared and set pH to 4-4.5 by the addition of glacial acetic acid. Container is covered with sterile paper and tied with rubber. The next day, as much as 20% (v / v) inoculum incorporated into the medium and incubated for 6 days at room temperature to form *nata* (Arviyanti and Yulimartani 2009).

Preparation of Dry Powder of BC

Sheet of *nata de cassava* is washed with water and then boiled to remove remaining bacteria. Furthermore, nata sheets soaked in a solution of NaOH 1% (w/v) at room temperature for 24 hours, followed by 1% acetic acid for 24 hours to neutralize its pH. Nata sheet that has been neutralized then washed with water and squeezed using a Büchner funnel and vacuum pump to form thin sheets that are still wet. This thin sheet is dried in the sun and then crushed in a *blender* to make a powder.

Synthesis of Cellulose Acetate

Acetylation of BC powder is done by modified procedure of Arifin (2004) dan Pasla (2006). 1.8 g of BC powder was weighed in a plastic bottle with double lid then added 100 mL of acetic acid. Bottle strongly shaken by hand for 1 minute, then shuffled back to the *shaker* for 20 minutes. After that, filtered by vacuum pump with a Buchner funnel and squeezed. The same filtering was repeated once more. Furthermore, the powder soaked in 50 ml of acetic acid in double lids bottle. After 3 hours, the powder re-filtered with vacuum pump and squeezed and then incorporated into a new container.

BC powder is then added to a solution of glacial acetic acid-H_2SO_4 with ratio 100:1 (10:0.1 mL) and stirred strongly. After that, with a ratio of 1:5 acetic anhydride was added with a pipette drops gradually while stirring with a stirring rod unti thickened and allowed to stand for 2 hours, starting from the addition of the firs drops of acetic anhydride. After the acetylation process is complete, the suspensior hydrolyzed with 2.4 mL of glacial acetic acid-distilled water (2:1) and stirred at the firs few minutes, then was allowed to stand for 30 minutes, starting from the addition o dilute acetic acid. Furthermore, the hydrolysis solution was centrifuged for 15 minute: at a speed of 4000 rpm. The resulting supernatant was then inserted into a beake containing 500 ml of distilled water and stirred as strong as possible with a magneti stirrer to form white flakes (CA)

CA fragments obtained with the filtered-vacuum Büchner funnel and washed with 1N $NaHCO_3$ until CO_2 gas bubbles disappear, then washed again with distilled water These neutral fragments are squeezed and then dried in an oven with a temperature of 50 ºC for 24 hours until the CA is completely dry. The resulting products were ther analyzed for CA water content and levels of acetyl.

Preparation of Membranes

Preparation of membranes made with modified procedures of Pasla (2006). The first phase, CA is dissolved in dry acetone solvent (according to the degree of acetylatior generated) using a magnetic stirrer. Polymer ratio and solvent used by 16% (w / v). The solution is then poured onto glass plates. Furthermore, the solution is printed by pressing and interesting solution using the stir bar to obtain a thin layer that sticks tc the glass and left for 15 minutes to evaporate the solvent. Cross section of glass was then soaked in water until a thin layer of membrane that sticks to the glass apart.

Membrane characterization

FTIR Analysis

FTIR analysis performed on powdered cellulose and cellulose acetate membranes. Each sample was prepared to form pellets and then placed in the sample holder.

SEM Analysis

CA membrane is cut into small then placed on the sample. Then, the sample was placed in a tube for the gold-coated surface for 20 minutes. Furthermore, samples that had been coated is placed in a SEM tool and look for the best magnification for surface morphology can be observed. Images obtained then photographed and stored.

3. Result and Discussion

The addition of sugar as a carbon source as much as 10%, 0.5% ammonium sulphate as a source of nitrogen, *A. xylinum* as *starter* as much as 20%, and setting the pH to 4 with the addition of glacial acetic acid is the optimum conditions needed to make *nata de cassava* from 1 L of liquid waste tapioca (Avriyani and Yulimartani 2009). The addition of sugar which is less than 10% do not provide enough carbon source so that disrupt bacterial metabolism and bacterial growth would be hampered, whereas the addition of sugar more than 10% will cause the media is too concentrated so that the bacteria work to be not optimum. Setting the pH to 4 aims to make the *A. xylinum* is superior compared with other bacteria in terms of obtaining nutrients from the media for its growth, thus preventing the growth of other bacteria that can disturb the growth of nata.

Nata de cassava (BC) obtained in this study is white with a thickness of about 1 cm (Figure 4). Before acetylation,, BC is soaked in NaOH 1% to swell the structure of cellulose fibers so it can open and reduce the intramolecular hydrogen bond. Immersion in 1% CH_3COOH is intended to neutralize the alkalinity of the structure of BC that has been purified. The structure of the swollen cellulose can increase the accessibility of-OH group on cellulose to acetylation reagent. BC then dried and crushed to obtain a brown powder. BC powder produced has a tight structure due to intermolecular hydrogen bonding of cellulose (Arifin 2004).

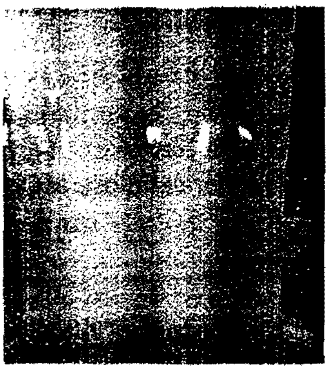

Figure 1 *Nata de cassava.*

Cellulose Acetate (CA)

Manufacture of cellulose acetate include four stages, namely activation, acetylation, hydrolysis, and purification. Stages of activation using concentrated acetic acid to attract water remaining in the powder BC because BC is hygroscopic powder. The process of immersion in acetic acid also increases the reactivity of cellulose as the BC matrix which is difficult to penetrate by chemical reagents will be swollen, so as to accelerate the penetration of acetic anhydride into the matrix at the time of acetylation.

Acetylation process intended to substitute BC hydroxyl groups with acet groups to form CA. Acetylation is an exothermic reaction, the temperature must b kept low to avoid degradation of the cellulose chain and to avoid evaporation (Windir and Hasche 1947). This can decrease the resulting yield will be reduced, or CA produc can not form at all. The addition of acetic anhydride dropwise followed by stirrir during the process of acetylation can maintain the solution temperature remained lo and avoid it, because the addition of acetic anhydride in significant quantities ca cause the system temperature to increase rapidly.

The third stage is hydrolysis, acetylation of the termination process with th addition of dilute acetic acid. Hydrolysis with dilute acetic acid can reduce the levels o acetyl substitution and reduce the degree of CA to the desired value. The reaction then purified by centrifugation to separate the acylated and BC are not. Parts of th acylated form of supernatant subsequently dispersed into distilled water to obtain tanned white flakes which is cellulose acetate (Figure 2).

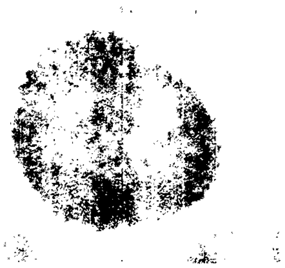

Figure 2 Powder cellulose acetate.

Cellulose Acetate (CA) Membranes

CA powder produced has a acetyl content of 40.38%. CA with acetyl content of 37 t 42% soluble in acetone solvent. Therefore, the CA powder produced in this study wer dissolved in acetone to make the CA membrane. Preparation of CA membrane made by phase inversion method. Polymer solution was printed on glass plates whose side are covered with tape.. CA solution was printed and allowed 15 minutes to evaporat the acetone until the solution becomes dry and form a membrane sheet. Membran sheets are then soaked in water to release it from the glass. The resulting membrane are white-brown (Figure 3).

Figure 3 Cellulose Acetate Membranes.

Water Levels and Levels of Acetyl CA

Levels show the abundance of acetyl-OH group in BC that has been acylated or the number of acetyl groups contained in the CA. . Factors that may affect the magnitude levels of acetyl cellulose is a method of drying, the concentration of NaOH mercerization on the stage or purification of cellulose, and cellulose ratio between weight and volume of acetic anhydride (Pasla 2006). The factors above have been optimized by Arifin (2004) and Yulianawati (2002).

Cultivated bacterial cellulose obtained containing low water levels due to moisture can affect the course of acetylation reaction. -OH group on the water more easily reacts with acetic acid anhydride as compared to the-OH groups on cellulose. Therefore, high accessibility to the cellulose-OH group is required to facilitate acetylation reaction by acetic acid anhydride reagents.. The higher accessibility of the cellulose-OH, acetyl levels would be even higher. CA acetyl content obtained in this study of 40.38%. . The value is equivalent to the levels of acetyl substitution degree of 2:51. The degree of substitution (DS) is the average number of H atoms on the hydroxyl group (-OH) is converted into acetyl group in each anhydroglucose residue. DS is associated with the application of CA in the industry. CA with DS 2.2 to 2.7 can be applied as a yarn and film.

CA with acetyl content of 40.38% can be dissolved in acetone (Kirk and Othmer 1993). Therefore, the solvent used to make the membrane CA in this study is acetone. According Wenten (1999), the higher the levels of acetyl, membrane pore size will be smaller and the water permeability will decrease. Based on the statement, the CA obtained in this study has pores small enough and low enough water permeability seen from the acetyl value which has high enough levels. This can be evidenced by the results of SEM analysis on the CA membrane surface that shows the range of membrane pore size (Fig. 6).

CA water content obtained was 21.49%. The amount of water content can be caused by the drying time of less CA long time in the oven so it has not gained the weight that is really constant. In addition, the cleanliness of the container and place of measurement can be influential because it can add weight to be weighed. The value of water content is necessary to calculate the levels of acetyl CA.

FTIR Spectra Analysis

FTIR spectrum analysis performed on cellulose *nata de coco, nata de cassava* powder, and CA. Cellulose *nata de coco* is the result of culturing the bacteria *A. xylinum* in coconut water medium without any additions.

In the FTIR spectrum of *nata de cassava* powder (Fig. 4) contained the absorption band at 3394.46 cm^{-1} wavelengths which is the stretching absorption band of-OH group as well as on the wavelength region 1059.15 cm^{-1} which is an absorption band stretching from the group CO. In addition, there are also absorption band for CH stretching in the wavelength region around 2900-3000 cm^{-1}. Uptake values are not much different also found on the FTIR spectrum of cellulose *nata de coco* (Fig. 5), ie 3367.85 cm^{-1} for-OH groups, 1059.21 cm^{-1} for CO groups, and around 2900-3000 cm^{-1} for CH stretching. This indicates that the constituent structure contained on *nata de cassava* powder is similar in structure to the cellulose constituent *nata de coco*. Therefore, based on the spectrum, *nata de cassava* powder can be regarded as cellulose, although there is still a possibility of other substances are impurities.

Figure 3 FTIR Spectra of Cellulose *nata de coco.*

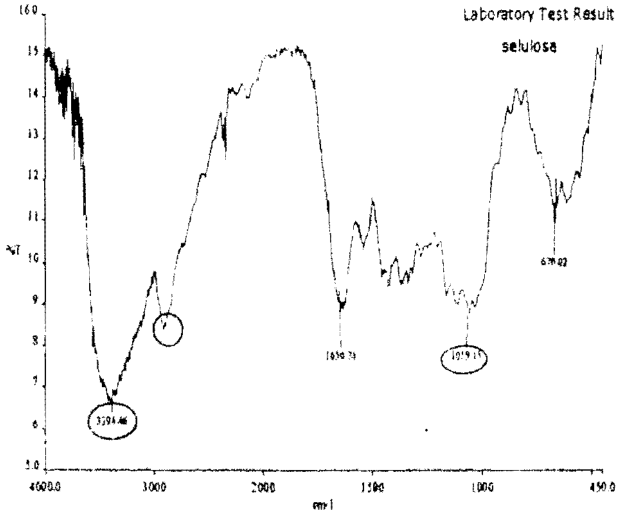

Figure 4 FTIR Spectra of Powder nata de cassava.

Nata de cassava powder spectrum compared with the membrane of FTIR CA to see the success of the acetylation process that has been done. In the FTIR spectrum of CA membrane (Fig. 5) there is a typical absorption band for carbonyl group or a C = O, ie the wavelength region 1754.81 cm $^{-1}$. In addition, there are absorption band for CO acetyl group at the wavelength of 1042.34 cm $^{-1}$. This indicates already the formation of acetyl groups in cellulose *nata de cassava* after diasetilasi to become CA. The existence of absorption band for-OH group at 3445.12 cm-1 region indicates that there is some residual hydroxyl groups on each anhidroglukosa are not acylated. This can be evidenced from the value of DS in CA that does not reach its maximum value, which is 3.

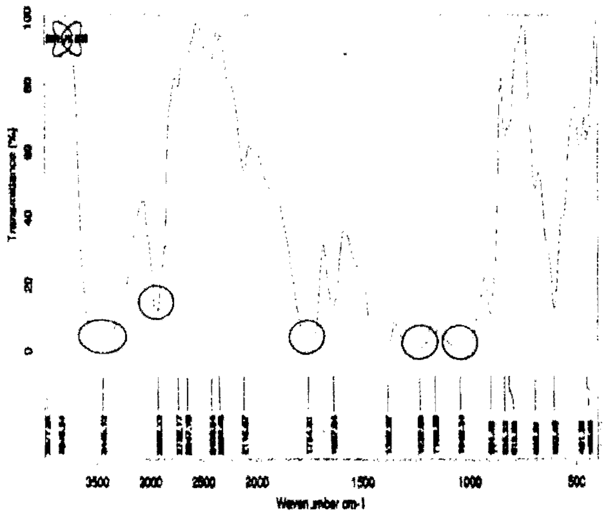

Figure 5 FTIR Spectra of CA Membranes.

Analysis of CA membranes by SEM

Characterization by SEM aims at observing the morphology of the surface membrane, namely by looking at the existence and size of membrane pores as well as latitude cross-section. From these observations, membrane type being analyzed be known. Observations made on two sections of the membrane, ie the surface and cross section latitude. The results of SEM with magnification of 5000x on the membrane surface showed the existence of pores that are spread on the membrane (Fig.6). The pore diameter of about 0.37-0.95 μm.. This suggests that the membrane can withstand the particles larger than 0.95 μm. in other words, permeate formed consisting of particle size ≤ 0.95 μm while rentetatnya be a particle size ≥ 0.95 μm. According to Mulder (1996), the membrane has a pore size of 0.1-10.0 μm classified as microfiltration membranes. Therefore, the resulting membrane CA is a microfiltration membrane.

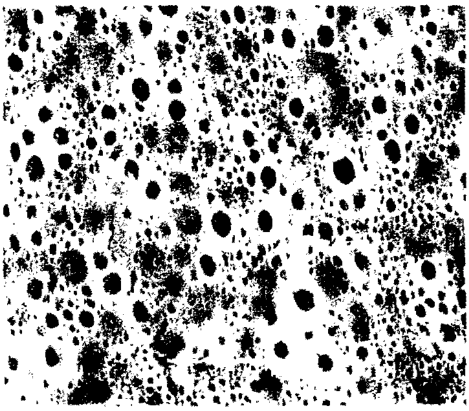

Figure 6 The Surface of CA Membrane With a Magnification of 5000 times.

Based on morphology, the observation of cross section of the membrane with a magnification of 2500 times (Fig.7) shows that the resulting membrane includes an asymmetric membrane as composed of several layers. Asymmetric membrane structure consists of layers of very dense and porous layers as a buffer. In asymmetric membranes, permeation occurs in a dense layer having a large mass transfer resistance (Lindu *et al* 2008).

Figure 7 Cross-section latitude CA membranes with 2500 times magnification.

5. Conclusion

Tapioca liquid waste can be made into nata de cassava. This is evidenced from the similarity spectrum of cellulose from nata de cassava with nata de coco. Cellulose nata de cassava can diasetilasi, then the resulting CA made into membranes by phase inversion technique using acetone solvent. CA formation is indicated by the results of FTIR analysis on CA membrane indicating the presence of C = O absorption band at wavenumber region 1725.92 cm-1 and CO acetyl at 1061.37 cm-1 region. CA has acquired the water levels of 21.49% and the acetyl content of 40.38% (equivalent to a range of degree of substitution of 2:51). SEM observations on CA membrane showed that the resulting membrane is a microfiltration membrane type and asymmetric.

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