

UTILIZATION OF ARECA NUT (*Areca catechu L.*) SHELL WASTE FOR CELLULOSE ACETATE MEMBRANES

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ABSTRACT

The skin of the betel nut contains 65.8% cellulose, the seeds of the fruit contain 53.20% cellulose fiber. The aim of the research is to synthesize cellulose acetate membranes from betel nut shell waste. The research method was cellulose isolation using soxhletation, bleaching, delignification and hydrolysis methods, synthesis of cellulose acetate and cellulose acetate membranes with variations of 9 and 10% PEG. The cellulose content of banana peel is 28.09% with β cellulose type and monoclinic crystal form. The tensile test results for PEG 9% were 1.4437 Mpa and PEG 10% were 1.2429 Mpa. The swelling test results of PEG 9% were 150.7% and PEG 10% were 196%. It was concluded that betel nut shell waste can be synthesized into cellulose acetate membranes.

Keywords: membrane; cellulose; cellulose acetate; PEG; areca nut

ABSTRAK

Kulit buah pinang mengandung 65,8% selulosa, biji buahnya mengandung serat selulosa 53,20%. Tujuan penelitian untuk sintesis membran selulosa asetat dari limbah kulit buah pinang. Metode penelitian dilakukan isolasi selulosa dengan metode sokletasi, bleaching, delignifikasi dan hidrolisis, sintesis selulosa asetat dan membran selulosa asetat dengan variasi PEG 9 dan 10%. Kadar selulosa dari kulit buah piang sebesar 28,09% dengan jenis β selulosa dan bentuk kristal monoklinik. Hasil uji tarik PEG 9% sebesar 1.4437 Mpa dan PEG 10% sebesar 1.2429 Mpa. Hasil uji swelling PEG 9% sebesar 150.7 % dan PEG 10% sebesar 196%. Disimpulkan limbah kulit buah pinang dapat disintesis menjadi membran selulosa asetat.

Kata kunci : membran, selulosa, selulosa asetat, PEG, kulit buah pinang

Introduction

Areca nut is one of Indonesia's vast natural resources, where the land area of areca nut plants in 2014 was around 137,000 ha with a production of 47,000 tons. (BPS, 2017). Based on this data, Indonesia is the largest producer of areca nut plants in the world. In 2018 Aceh Province became one of the largest areca nuts producing provinces in Indonesia reaching 40,680 ha with a production of 15,488 tons. The production was

spread in the districts of North Aceh, Bireuen and East Aceh with a production of 4,264 tons, 3,710 tons and 1,621 tons (BPS Aceh, 2020).

The utilization of areca nut seeds in the community is generally as a food coloring. Due to the utilization of only areca nut seeds, the skin of areca nuts is discarded and becomes waste. The waste from the areca nuts shell has no further handling. In addition, people also often burn areca nut shell waste, causing problems for the environment, namely causing air pollution (Rosalina & Febriadi, 2019). However, areca nut shells have benefits as liquid organic fertilizer and filters in making bioplastics. The utilization of areca fruit peel is because it has high cellulose fiber (Ramadhani Tamiogy et al., 2019).

Areca nuts contain chemical compounds consisting of fats, polyphenols (flavonoids and tannins), alkaloids and minerals. In addition, areca nuts also contain cellulose. The areca nut skin contains 65.8% cellulose, while the seeds contain 53.20% cellulose fiber (Kencanawati et al., 2018). Ramadhani Tamiogy et al. (2019) mentioned that the cellulose content in the areca nut skin is around 34.18%, then 20.83% hemicellulose and 31.6% lignin. The different cellulose content is due to the location of growth and cultivation. The components in plants are composed of almost 60% of cellulose which is part of carbohydrates (Azizah & Marziah, 2018), so cellulose can be used as raw material for making fabrics, bioethanol (Dewanti, 2018), bioplastics (Ramadhani Tamiogy et al., 2019), papermaking, biodegradable plastics and membranes (Kentjana et al., 2008).

The development of renewable technology is currently one of the world's centers of attention. Renewable technology that is growing rapidly and is of concern is separation technology using membranes made from natural polymers (Nurman et al., 2015). Separation technology using membranes is one alternative method that is widely practiced as a substitute for conventional separation technology (Apriani et al., 2017). Membrane technology is used because it has advantages, including low energy required, the process is carried out sustainably, can be combined with other methods, and does not produce waste (Yave et al., 2010). Membrane technology that has been synthesized includes polyurethane (Nurman et al., 2015; Nisah, et al, 2022; Marlina, 2012), chitosan (Rohman et al., 2009; Berghuis et al., 2020) and cellulose acetate (Safiah & Mulyati, 2018; Harianingsih & Maharani, 2018).

Cellulose acetate membrane is one type of membrane technology based on biopolymers from cellulose acetate (Apriani et al., 2017). The advantage of using this cellulose acetate membrane is the easy production process and raw materials from renewable natural resources (Iriyanti, 2016). Cellulose acetate membranes can be synthesized from water hyacinth (Rachmawaty et al., 2013) and empty palm bunches (Apriani, 2018). Cellulose acetate as a membrane requires solvents and plasticizers including acetone, normal methyl pyrrolidone (NMP), di methyl acetamide (DMAc), dimethyl formamide (DMF) and polyethylene glycol (PEG) (Ahmad, 2005). The use of polyethylene glycol (PEG) is due to its non-toxic, biocompatible, hydrophilic, high flexibility, antifoaming agent, and antifouling properties. The addition of PEG to the membrane can produce membranes with smaller and more organized pores and can increase the flux value of the membrane (Muliawati, 2012). Cellulose acetate as a membrane is obtained from processing cellulose from areca nut shells through a hydrolysis process using strong acids (HCl, HNO₃ and H₂SO₄) with the aim of

breaking bonds and crystal structures from lignocellulose. (Maryam et al., 2019). The process of processing cellulose from areca nut peel has been widely done, as done by researchers from India Chandra et al., (2016) using the soxhletation method with toluene solvent: ethanol (2:1) obtained α -cellulose content of 34.18%. Soman et al. (2017) also extracted areca nut peel using the soxhletation method with a time of 6 hours and made a solvent comparison of toluene: ethanol (2:1) obtained α -cellulose content of 60%. Research conducted by Sultana et al., (2020) also extracted areca nut peel from the fruit Bangladesh with maceration method for 10 hours obtained α -cellulose content of 51.08%.

Based on the above explanation, the type and content of cellulose from areca nut peels obtained from Aceh Province were analyzed and then cellulose acetate membrane was synthesized. Cellulose characterization using X-Ray Diffraction (XRD) and FTIR spectroscopy. Acetylation process of cellulose acetate membrane with the ratio of cellulose acetate, acetone and the addition of PEG varied at 9% and 10%. Characterization of cellulose acetate membrane from areca nut peel using Scanning Electron Microscopy (SEM), Tensile and Swelling test.

Research method

Tools and Materials

Tools

The tools used in this research are a set of centrifugation tools, a set of centrifugation tools, XRD (X-Ray Diffraction) Shimadzu type XRD-7000 MAXima, Scanning Electron Microscopy (SEM) JSM-6360 instrument, glassware, water bath, analytical balance, hot plate, 100 mesh sieve, magnetic stirrer, blender, drying oven, universal pH indicator paper, thermometer and watch glass.

Materials

The materials used in this study were toluene (C₇H₈) 98%, ethanol (C₂H₅OH) 99%, sodium hydroxide (NaOH) 5% (b/v), hydrochloric acid (HCl) 2 N, sulfuric acid (H₂SO₄) 1 N and 72% (v/v), sodium hypochlorite (NaOCl) 5% (v/v), distilled water (H₂O), Benedict's reagent, iodine reagent, Acetate an-hydrate ((CH₃CO)₂O), Glacial acetic acid (CH₃COOH), Acetone (CH₃COCH₃), Sulfuric acid (H₂SO₄), Polyethylene Glycol (PEG) 400, and *Areca catechu* L. fruit peel.

Work Procedure

Sampling

The areca nut shells were taken from North Aceh District from one areca nut-growing village, thus representing all villages in the district.

Sample Preparation

Areca nut bark was washed and dried for 2-3 days, cut into 1-2 cm pieces, pulverized with a blender and dried in an oven at 85 °C for 24 hours to a coarse powder. The coarse powder was filtered with a 100 mesh sieve (Tamiogy et al., 2019).

Cellulose Isolation

Extraction of areca nut peel powder was carried out using the succulent method. 50 g of areca nut shell powder was dissolved with a mixture of toluene: ethanol (2:1) solvent for 6 hours. The residue was bleached by immersing it in 300 mL of 5% NaOCl at 60°C for 3 hours to remove lignin. Then filtered and washed with distilled water until neutral residue (pH = 7). Then proceed to the delignification process.

To remove hemicellulose and pectin, the residue was then added with 400 mL of 5% NaOH at 80°C for 2 hours, filtered, washed with distilled water until alkali-free and dried. Furthermore, the hydrolysis process was carried out with strong acid treatment of HCl 2 N. The hydrolysis process was carried out by adding the residue of delignification and bleaching results with 500 mL of HCl 2 N then stirring with a magnetic stirrer for 30 minutes at room temperature. Then filtered, washed until the pH of the filtrate reaches 7 and dried at 50°C for 24 hours then weighed the final weight (Soman et al., 2017 and Dewi et al., 2017). Calculate the yield. The extract was analyzed qualitatively in the form of iodine and Benedict's tests, as well as FTIR. Quantitative analysis was carried out to determine cellulose content using the Chesson method. After calculating the cellulose yield, it was analyzed by X-Ray Diffraction (XRD), from the X-Ray Diffraction (XRD) analysis, the degree of crystallinity and the size of cellulose crystals can be known. Crystal size measurement is done using the Scherrer equation.

Analysis of cellulose content used the Chesson method:

- a) A total of 1 g of dry sample (A) was added to 150 mL of distilled water, refluxed at 100°C with a water bath for 1 hour. The result was filtered, the residue was washed with hot water (300 mL). The residue was then oven dried until constant and then weighed.
- b) The residue was added 150 mL of 1 N H₂SO₄ then refluxed with a water bath for 1 hour at 100°C. The result was filtered and washed with distilled water until neutral (300 mL) then dried and weighed (B)
- c) The dried residue was added to 10 mL of 72% H₂SO₄ and soaked at room temperature for 4 hours. Then 150 mL of 1 N H₂SO₄ was added and refluxed on a water bath for 1 hour on counter cooling. The residue was filtered and washed with distilled water until neutral (400 mL) then heated in an oven at 105°C and the results were weighed (C) (Aprilyanti, 2018).

Synthetic cellulose acetate

Cellulose acetate synthesis refers to the method of (Bahmid et al, 2014) which has been modified by (Apriana et al., 2018). A total of 15 g of cellulose was added to 150 ml of glacial acetic acid in a ratio of 1:10 and stirred using a magnetic stirrer for 60 minutes at 38 °C. Next, 2% (v/v) H₂SO₄ was added to the solution and stirred for 45 minutes at 38°C. The activation results were added to 100mL of acetic acid anhydride then stirred at 38°C and within 45 minutes. The acetylated product was added 10 mL H₂O and 20 mL H₂SO₄ and stirred for 30 minutes at 50°C. The solution was centrifuged. The resulting precipitate from the centrifugation process was put into 500mL of distilled water to form cellulose acetate flakes. Followed by screening or separation of cellulose acetate powder with distilled water through a vacuum filtration

process. The wet cellulose powder obtained was put into the oven for 4 hours at 55°C and obtained cellulose acetate powder (Vashti et al., 2024).

Synthetic cellulose acetate membrane

The membrane was prepared by mixing 4 g of cellulose acetate and 80 mL of acetone in a ratio of 1:20 % by weight into a closed Erlenmeyer flask and stirred for 33 hours with periodic stirring at room temperature until homogeneous (Malik, 2016). Then the solution was added with PEG 9% and 10% as much as 40 mL and stirred again for 22 hours with periodic stirring. The solution was left for several hours to remove air bubbles. The well-mixed solution was poured on a glass plate and left at room temperature so that it was printed into a thin film. The formed membrane was washed with running water for several hours to remove all solvents and additives (Apriana et al., 2018). The membrane was stored in 1 ppm sodium azide solution to prevent microbial growth (Radiman & Eka, 2007). Cellulose acetate membranes were characterized by Scanning Electron Microscopy (SEM), Tensile Test, and Swelling Test.

Results and discussion

The results of the study explained that the qualitative test of cellulose in areca nut (*Areca catechu* L.) fruit peel obtained data which can be seen in Table 1.

Table 1. Qualitative Test of Areca Peel Cellulose

No	Qualitative analysis	Result
1	Benedict's test	(+) Green color precipitate
2	Iodine Test	(+) Brown color precipitate

Benedict test is conducted to determine the content of reducing sugars (carbohydrates) in a sample (Hani et al., 2023). In the Benedict test, the sample is declared to contain cellulose indicated by the formation of a brick red precipitate which is then colored green, red or orange (Hani et al., 2023). The principle of this benedict test is that free aldehyde or ketone groups on reducing sugars contained in the sample reduce Cu^{2+} ions from $CuSO_4 \cdot 5H_2O$ in an alkaline atmosphere to Cu^+ which precipitates into Cu_2O . Alkaline atmosphere is obtained from Na_2CO_3 and sodium citrate contained in benedict reagent (Kusbandari, 2015).

The results of the iodine test show positive when a brown color is formed and belongs to the type of polysaccharide (Hani et al., 2023). The iodine test is carried out to identify the type of polysaccharide. The results of the iodine test inform that each type of polysaccharide will give the results of complex compounds with different colors such as blue test results indicating positive formation of amyllum compounds, red-brown color produces glycogen compounds and the addition of iodine to cellulose suspensions produces brown complex compounds (Fitri & Fitriana, 2020).

Cellulose is defined as a very long and unbranched polymer (straight chain) of 1,4-β-D-glucose (Annisa et al., 2018). The results of XRD diffractogram analysis provide information related to the polymer structure of cellulose which is in the amorphous and crystalline regions, this is because cellulose has a crystalline structure while hemicellulose and lignin have an amorphous structure (Trisanti et al., 2018).

Qualitative observations revealed that the cellulose structure analyzed was a mixture of crystalline and amorphous structures (Figure 1).

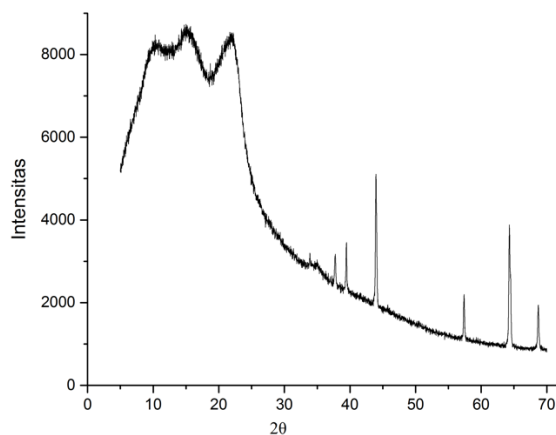


Figure 1. XRD diffractogram of areca nut shell cellulose

Further analysis was carried out from the XRD characterization data to determine the type of cellulose that was successfully obtained from the extraction process by soxhletation, obtained peaks on the XRD diffractogram of cellulose with high intensity at 2θ angles of 21.52° , 15.02° and 10.40° . The peak broadening is mostly due to the presence of non-cellulose materials such as hemicellulose and lignin present in the sample (Suryanto, 2017). At the peak of $2\theta = 21.52^\circ$, the distance between diffraction planes for the miller index (210) was 4.12415 \AA with a crystallinity index of 96.99% and a degree of crystallinity of 97.07%. Nurlia et al., (2020) explained that the greater the percentage of crystallinity degree, the higher the peak of the diffractogram, thus negating the amorphous nature. The high value of crystallinity is due to the hydrolysis treatment process using hydrochloric acid solvent. The use of this acid solvent can eliminate the amorphous nature of the cellulose chain (Febrianto & Nikmatin, 2013).

Figure 1 describes that the crystals formed experience overlapping or buildup, while to obtain perfect cellulose crystals the resulting peak must be sharp. Peaks that experience overlapping so that they can be analyzed are carried out deconvolution or fitting using the Gaussian equation. Figure 2 presents the results of Gaussian fitting of cellulose XRD data. After being matched with the data in JCPDS No. 03-0226, the 2θ angle positions at 14.44° ; 16.03° and 20.22° were obtained.

The shape of cellulose crystals was calculated using Match software. The results obtained that cellulose from areca nut shell is in the form of monoclinic crystals with $a = 0.9056 \text{ nm}$, $b = 1.026 \text{ nm}$, $c = 1.018 \text{ nm}$ and $\alpha = \gamma = 90.0^\circ$, $\beta = 96.750^\circ$, because the dimensional value of $a \neq b \neq c$ $\alpha = \gamma = 90.0^\circ$, $\beta \neq 90^\circ$ is characteristic of monoclinic crystals (Nurjannah et al., 2020). The results of this calculation are almost the same as the Joint Committee on Powder Diffraction Standards (JCPDS) database No. 03-0226 (cellulose) which has a dimension value of $a = 0.787 \text{ nm}$, $b = 1.031 \text{ nm}$, $c = 1.013 \text{ nm}$. Suryanto, (2017) who examined bacterial cellulose obtained a crystal form that is triclinic with cell dimensions $a = 0.674 \text{ nm}$, $b = 0.593 \text{ nm}$, $c = 1.036 \text{ nm}$ (chain axis), $\alpha = 117^\circ$, $\beta = 113^\circ$, $\gamma = 81^\circ$. The results of JCPDS data synchronized with standard

data can be obtained from cellulose from areca nut peel including β cellulose type supported by monoclinic crystal shape.

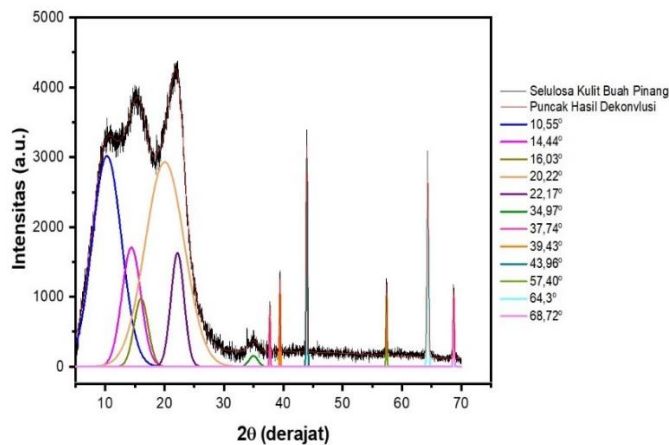


Figure 2. Gaussian Fitting Results of Cellulose XRD Data

Quantitative analysis was also conducted to determine cellulose content. Quantitative analysis was carried out using the Chesson method. The cellulose content produced by the Chesson method can be seen in Table 2. The cellulose content produced from areca nut peel was 28.09% while the % yield obtained was 36%. Cellulose content shows the purity of cellulose from the extraction results while the yield shows how much cellulose extract is obtained.

Based on the results of the quantitative test of cellulose in areca nut (*Areca catechu* L.) peel, the following data were obtained:

Table 2. Quantitative Test of Cellulose in Areca Peels

Sample	Cellulose Content (%)	Cellulose Yield (%)
Areca nut peel (<i>Areca catechu</i> L.)	28,09	36

Research conducted by Soman et al. (2017) on areca fruit peel from India using the soxhletation method obtained α -cellulose content of 60%, while research conducted by Sultana et al. (2020) on areca fruit peel obtained from Bangladesh produced α -cellulose content of 51.08% using the maceration method. The difference in cellulose content from different regions is due to the location of growth and cultivation of plants, solvents used during extraction and extraction methods.

FTIR Spectrophotometer Analysis

To identify cellulose from areca nut peel, characterization was carried out using FTIR spectroscopy. FTIR characterization aims to identify cellulose functional groups. Absorption at wave number $3273\text{ cm}^{-1} - 3591\text{ cm}^{-1}$ the presence of O-H group stretching vibrations. The C-H functional group is found at wave number 2937.59 cm^{-1}

1 and the C-O group at wave number 1265 cm⁻¹ and the C-O-C group is at wave number 1060.85 cm⁻¹. In addition to these groups, the 1300 cm⁻¹ fingerprint area indicates the presence of C-H bending and around 1400 cm⁻¹ indicates the bending vibration of CH₂ (Riani et al., 2019). The results obtained show that the O-H, C-H and C-O and C-O-C functional groups are the main groups of cellulose (Ischak et al., 2021). The FTIR (Fourier Transform InfraRed) spectrum of areca nut (*Areca catechu* L.) peel can be seen in image 3.

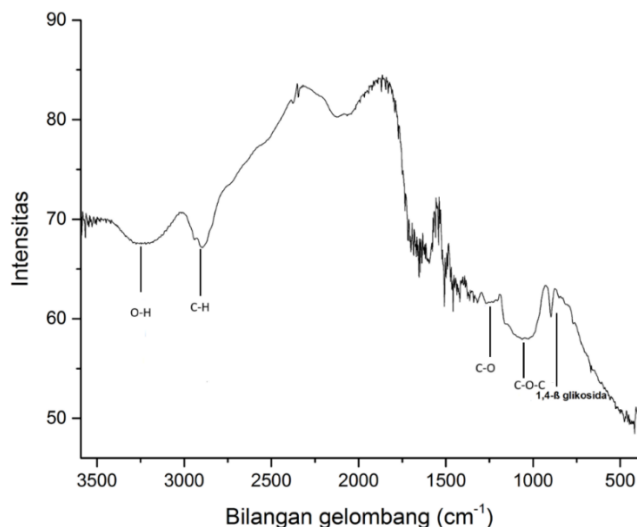


Figure 3. FTIR spectrum of cellulose from areca nut shells

Cellulose acetate showed the absorption of C=O carbonyl group and C-O-C ester group at wave numbers 1749-1622 cm⁻¹ and 1370.49 cm⁻¹ (image 4).

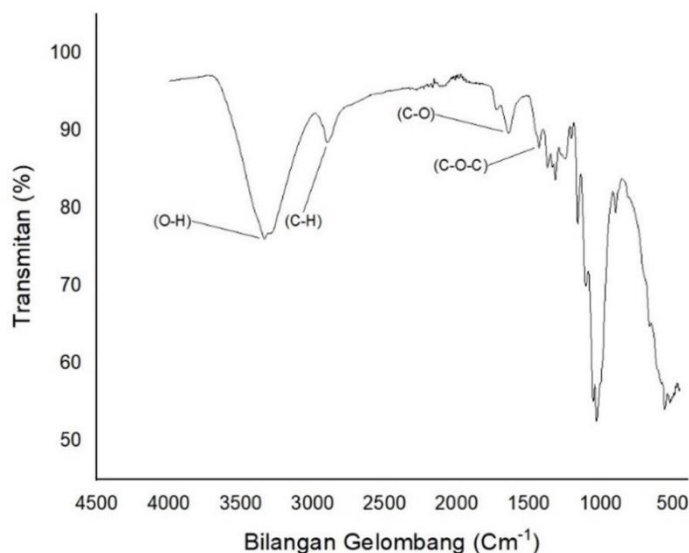


Figure 4. FTIR spectrum of cellulose acetate from areca nut shells

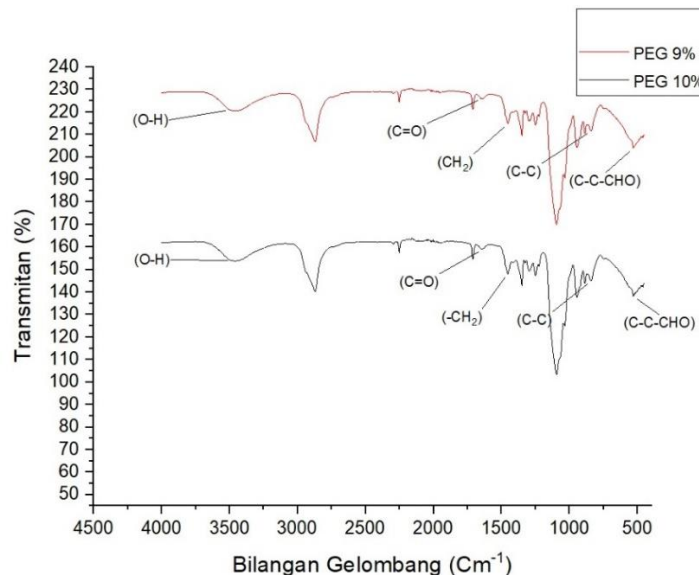


Figure 5. FTIR spectrum of cellulose acetate membrane from areca nut peel

Characterization of cellulose acetate membrane using FTIR obtained typical absorption peaks for cellulose acetate characterized by the appearance of -OH group at 3465.31 cm⁻¹ for PEG 9% variation and the same result for PEG 10% variation, carbonyl group (C=O) at 1643.16 cm⁻¹ in PEG 9% variation and 1643.45 cm⁻¹ in the variation of PEG 10%, the absorption of the -CH₂ group obtained an absorption of 1453.14 in the variation of PEG 9% and 1453.14 cm⁻¹ in the variation of PEG 10%, reinforced by the absorption of the C-C group which obtained an absorption of 885.93 in the variation of PEG 9% and obtained 885.52 in the variation of PEG 10%, and obtained the C-C-CHO group of 530.80 in the variation of PEG 9% and 530.72 in the variation of 10%. Research conducted by Muliawati, (2012) the manufacture and characteristics of nanofiltration membranes for water treatment states that the appearance of typical absorption of cellulose acetate is characterized by the appearance of OH groups at 3417.4 cm⁻¹. The appearance of acetyl groups is characterized by the absorption of carbonyl groups C=O and -CH₂ at wave numbers 1643.9 cm⁻¹ and 1438.4 cm⁻¹ (Figure 5). This proves that cellulose acetate was successfully synthesized into cellulose acetate membrane.

Characterization of cellulose acetate membrane from areca nut shell waste

The results of Scanning Electron Microscopy on cellulose acetate membranes from areca nut shell waste cellulose can be seen in Figure 6. 9% PEG variation with 5000x surface magnification (A) shows uneven holes or cavities and gets a membrane size of 2 μm. While the 10% PEG variation with a cross-sectional magnification of 5000x (B) looks like there are cracks but there are many open cavities and get a pore size value of 3 μm, cracks can be caused when removing the membrane from the glass plate. Based on the pore size obtained in both membrane variations, the membrane produced in this study is a microfiltration membrane category.

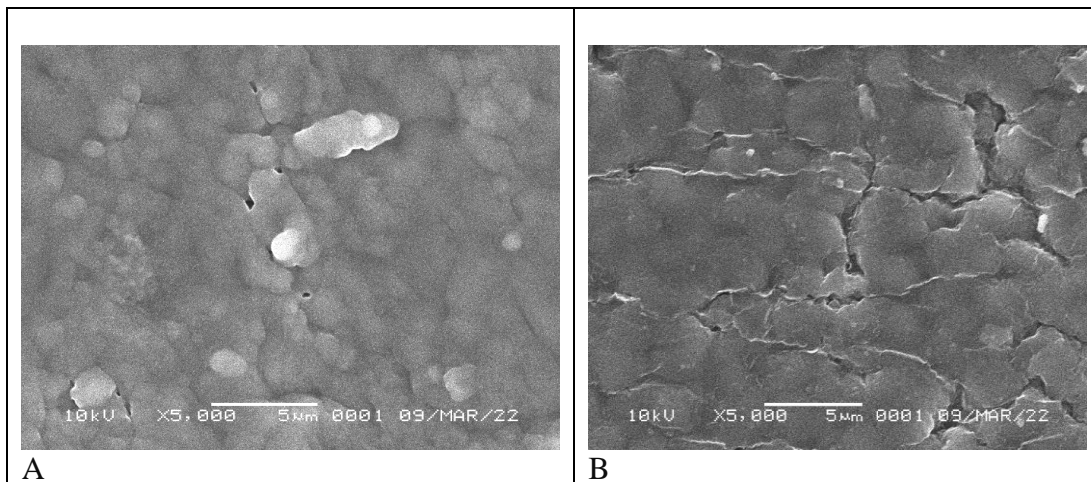


Figure 6. SEM test results on cellulose acetate membranes from areca nut peel waste cellulose with 9% PEG variation (A) magnification of 5000x and 10% PEG variation (B) magnification of 5000x.

Tensile test

The tensile test explains the elasticity of a membrane and the strength of a membrane when given a force or pressure (Izzaty et al., 1967). The tensile test is conducted to determine how strong the membrane withstands the force or pressure applied to the membrane so that the membrane is not easily damaged. Membranes that have a strong tensile strength are membranes that have a high level of inter-pore density (Azzahra, 2022). The tensile strength of cellulose acetate membrane can be seen in Table 3.

Table 3 Tensile Test Results of Cellulose Acetate Membrane

Test Parameters	Units	Variation	Test result
tensile strength	MPa	PEG 9%	1.4437
tensile strength	MPa	PEG 10%	1.2429

The tensile test results of PEG 9% amounted to 1.4437 Mpa and PEG 10% amounted to 1.2429 MPa. The value at PEG 9% is higher than the value obtained by PEG 10%, this is because the more PEG that is added, the more membrane pores are formed, these pores result in the formation of spaces in the membrane so that the membrane has a tenuous structure and its tensile strength weakens (Izzaty et al., 1967). If PEG has a high concentration, it can result in a decrease in the mechanical strength of the membrane. Membranes made using the right PEG concentration will make the membrane dense, strong and good structure (Azzahra, 2022).

Swelling Test

Table 4. Cellulose Acetate Membrane Swelling Test Results

Membrane	Variation	initial weight	final weight	Swelling Degree
MembraneCellulose Acetate	PEG 9%	0.2068	0.5186	150.7 %
MembraneCellulose Acetate	PEG 10%	0.2071	0.6131	196 %

The results of the swelling test can be seen in Table 4 where the test results with the addition of PEG 9% amounted to 150.7% and PEG 10% amounted to 196%. The swelling test is carried out to predict the size of the substance that can be diffused by the cellulose acetate membrane and also indicates the presence of cavities that exist between polymer bonds (Amanda, 2022). The results showed that the membrane with 9% PEG variation diffused less substances because the cavities were very tight or very little.

Conclusion

Areca nut peel was positive for cellulose as indicated by iodine test and Benedict test. The cellulose content of areca fruit peel is 28.09% with β cellulose type and monoclinic crystal form. The absorption at wave numbers 3273 cm^{-1} - 3591 cm^{-1} is the stretching vibration of the O-H group. The C-H functional group is at wave number 2937.59 cm^{-1} and the C-O group at wave number 1265 cm^{-1} and the C-O-C group is at wave number 1060.85 cm^{-1} . Cellulose acetate showed the absorption of C=O carbonyl group and C-O-C ester group at wave numbers 1749-1622 cm^{-1} and 1370.49 cm^{-1} . The -OH group at 3465.31 cm^{-1} for the PEG 9% variation and the same result for the PEG 10% variation, the carbonyl group (C=O) at 1643.16 cm^{-1} in the PEG 9% variation and 1643.45 cm^{-1} in the PEG 10% variation, the absorption of the -CH₂ group obtained an absorption of 1453.14 in the PEG 9% variation and 1453.14 cm^{-1} in the PEG 10% variation, reinforced by the absorption of the C-C group which got an absorption of 885.93 in the PEG 9% variation and 885.52 in the PEG 10% variation, and obtained the C-C-CHO group of 530.80 in the PEG 9% variation and 530.72 in the 10% variation. The tensile test results of PEG 9% amounted to 1.4437 Mpa and PEG 10% amounted to 1.2429 Mpa. The swelling test results of PEG 9% addition amounted to 150.7% and PEG 10% amounted to 196%.

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