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Preparation and Characterization of Cellulose Acetate from *Pandanus tectorius* via Microwave Irradiation

Meta Fitri Rizkiana*, Abdul Malik Karim Amrullah, Nor Elisah, Helda Wika Amini, Bekti Palupi, Istiqomah Rahmawati, Boy Arief Fachri

Chemical Engineering Department, Research Center for Biobased Chemical Product, Faculty of Engineering, Universitas Jember

*Corresponding Author: metafitririzkiana@unej.ac.id

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Abstract

This research focused on extracting cellulose from screw pine and evaluating its potential as a raw material for cellulose acetate. Screw pine contains polysaccharides, especially cellulose. In its fabrication process, cellulose acetate was produced from the cellulose acetylation reaction. Cellulose was isolated using Microwave-assisted Extraction with variations in NaOH concentration (1, 2, and 3%), H_2O_2 concentration (10, 20, and 30%), and time variables (20, 40, and 60 minutes)—acetylation using Anhydrous Acetic Acid and H_2SO_4 catalyst. Extraction optimization was performed with the Response Surface Methodology (RSM) model BBD (Box-Behken Design) with 17 runs. Several characterizations were carried out to determine the characteristics of cellulose acetate, namely Degree of Acetylation, FT-IR spectroscopy, and X-ray diffraction (XRD). The highest cellulose yield obtained in the extraction process was 50.7% with a variable of 3% NaOH, 30% H_2O_2, and 60 minutes. XRD analysis showed a peak at $2\theta = 22.53798°$ similar to commercial cellulose. FTIR functional group identification of cellulose acetate showed the presence of carbonyl (C=O) and (C-O Acetyl) peaks, seen at wavenumbers 1734 cm^{-1} and 1256 cm^{-1}. The acetyl content of cellulose acetate from screw pine produced was 35.475%. Therefore, screw pine leaves have the potential to be a raw material for cellulose acetate production.

Keywords: Extraction, Microwave-Asisted, Cellulose Acetate, Pandanus tectorius Leaves

INTRODUCTION

Indonesia is rich in natural resources, so many biomasses can be converted into various applications such as fuel, biomaterials, fertilizers, and pharmaceuticals. One of the biomasses utilized is screw pine leaves or commonly referred to as mengkuang (Esa, 2011). Screw pine is usually found around beaches and swamps. The tensile strength of fibers in screw pine leaves is proportional to the cellulose content (Hamizol & Megat-Yusoff, 2015). Good strength of screw pine fiber is contributed from high cellulose content 73.10% and low wax content 0.35% (Selvan et al., 2022). Utilization of screw pine leaves in Indonesia is limited to handicrafts marketed in the Asian region. Research related to the composition and cellulose isolation of screw pine leaves still needs to be developed (Sheltami, Abdullah, Ahmad, Dufresne, & Kargarzadeh, 2012). The screw pine leaves as shown in Figure 1.

Figure 1. Screw pine Leaves

Cellulose is found in the form of lignocellulose, along with lignin and hemicellulose (Mulyadi, 2019). Cellulose is a natural, biodegradable, and renewable polymer that can be utilized as construction raw material, adsorption material, insulating material, biomedical applications, composites, membranes, and eco-friendly products. In recent decades, cellulose has received particular attention in biomass-based research. This is due to the nature of cellulose as a biopolymer with a production capacity of 10^9-

1.5×10^{12} tons/year (Nechyporchuk, Belgacem, & Bras, 2016). Cellulose is the raw material of cellulose acetate, commonly used in the industry. Cellulose acetate is cellulose with acetyl groups replacing hydroxyl groups (Souhoka & Latupeirissa, 2018). Cellulose acetate is a cellulose-derived compound that goes through an acetylation process with the help of a reagent in the form of acetic anhydride (Syamsu & Kuryani, 2014). Cellulose acetate is solid, odorless, and white (Yulandri, 2020). Cellulose acetate is widely used in filtration processes, membranes, biofilms, plastics, sensors, and coating materials. In addition, cellulose acetate is utilized in textiles, pharmacology, medicine, chromatography, and waste treatment (Souhoka & Latupeirissa, 2018).

Before the cellulose acetate fabrication process, extraction must be done first to isolate cellulose. The extraction process could be done sequentially by cleaning from impurities, alkali treatment, filtration, bleaching, and drying (Elias, 2014). The following data are previous research that discusses the extraction of cellulose from various biomasses can be seen in Table 1:

Table 1. Previous research on the extraction of cellulose from various biomasses

Type of Biomass	Method	Optimum condition	Yield (%)	Source
Screw pine Leaves	Alkali NaOH 10%	Time : 120 minute Temperature : 170°C	71.54 32 %	(Hamizol & Megat-Yusoff, 2015)
Screw pine Leaves	Alkali NaOH 6%	Time : 60 minute Temperature : 170°C	-	(Elias, 2014)
Screw pine Leaves	Alkali NaOH 25%	Time : 4 hours Temperature : 170°C	88.27 %	(Yanti, Syafii, Wistara, & Febrianto, 2018)
Screw pine Leaves	Alkali NaOH 4% and $NaClO_2$ 1,7%	Time : 2 hours Temperature : 125°C Bleaching process Time : 4 hours Temperature : 125 °C	57.5 %	(Sheltami et al., 2012)
Banana Stem	Microwave NaOH 2,9% and H_2O_2 15,10%	Time : 21,29 minute Power : 450 watt Bleaching process Time : 1 hours	82.13 6%	(Thi et al., 2022)
Hemps stalk	Microwave NaOH 2.5 M and H_2O_2 30%	Time : 45 minute Power : 350 watt Temperature : 90°C Bleaching process Time : 4 hours Temperature : 55°C	84.32 %	(Chowdhury & Hamid, 2016)

The conventional extraction process consumes many chemicals, takes a longer time, and has a low yield (Maleta, Indrawati, Limantara, Hardo, & Brotosudarmo, 2018). The conventional process can also cause degradation of compounds during the extraction process (Kunaro, Sutardi, Supriyanto, & Anwar, 2019). However, the MAE method is more time-efficient and yields higher. Microwave extraction uses microwave assistance to heat a solvent in contact with samples and escalate mass transfer (Chowdhury & Hamid, 2016). The extracted cellulose increased proportionally to the extraction time and high NaOH concentration (Hamizol & Megat-Yusoff, 2015). The extraction process using microwave assistance and the continued bleaching process can increase the cellulose content produced, while the hemicellulose and lignin content is shallow (Thi et al., 2022).

Cellulose obtained through the extraction process is applied to synthesize cellulose acetate. The acetylation process in manufacturing cellulose acetate involves acetyl groups replacing hydroxyl groups on cellulose (Yulandri, 2020). The reaction of cellulose formation into cellulose acetate can be seen in Figure 2.

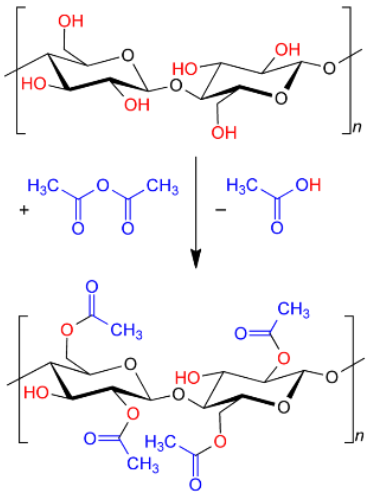

Figure 2. General reaction of cellulose acetate formation

Preparation of cellulose acetate with raw materials of nata powder and glacial acetic acid in a ratio of 1:5 produced acetyl content between 30.60-44.51% through the acetylation process for 1-3 hours at 38℃, the optimal time for acetylation process was 2 hours which can produce high acetyl content (Syamsu & Kuryani, 2014). The process of utilizing cellulose to cellulose acetate through an acetylation reaction required 2.5 hours at 25℃ and 5 hours at 40℃ using commercial α-cellulose raw materials (Sigma, CAS Number 9004-34-6) to obtain cellulose acetate of 1.4822% and 2.295% respectively and acetyl content of 28.413% and 38.2017% (Purwanti, Luliana, & Sari, 2018). Synthesis of cellulose acetate from banana leaf raw materials with an acetylation time of 1.5 hours at 38℃ was obtained with cellulose acetate content of 50% (Purwanti et al., 2018).

This research aims to investigate optimum cellulose extraction condition of screw pine with parameters such as raw material (NaOH and H_2O_2 concentration and time. This research fabricates cellulose to cellulose acetate, which has a high commercial value. This study determines the characteristics of cellulose and cellulose acetate through Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD).

METHODOLOGY

Materials and Instrumentals

Screw pine leaves were obtained from Papuma Beach in Wuluhan District, Jember Regency, East Java. Other materials used include Anhydrous Acetic Acid, Glacial Acetic Acid, Aquadest, H_2O_2 (10, 20, and 30%), H_2SO_4, NaOH (1, 2, and 3%), HCl, and PP Indicator. Extraction was carried out using a microwave with a power of 300 watts.

Pre-treatment Methods

The screw pine leaves were separated from their thorns. The leaves were cut to 10 cm to ensure an effective drying process and then dried in the sun for 24 hours. This drying aims to reduce the water content in the leaves and turn them into a more durable dry form (Purwanti et al., 2018). The dried leaves were soaked with water for three days, and the soaking water was changed frequently to remove odors and soften the dried leaves. The soaked leaves were boiled with water for 15 minutes; then, the leaves were washed several times with water. The leaves were dried in a 60°C oven and measured for moisture content. The material was then pulverized using a blender and sieved with an 80-mesh size. After the pre-treatment process, the water content contained in screw pine leaves was measured. The water content can be calculated as follows:

$$\text{Water content} = \frac{(\text{initial mass (g)} - \text{final mass (g)})}{\text{initial mass (g)}} 100\% \quad (1)$$

Cellulose Isolation

Extraction was carried out after the pre-treatment stage. The resulting material was treated with alkali NaOH. NaOH solvent is used in alkaline extraction to reduce lignin content and improve cellulose quality (Yulandri, 2020). NaOH was used with variations (1, 2, and 3%) with the ratio of material: solvent 1:20. The choice of solvent is also one of the critical factors of the extraction process (Dewatisari, 2020). The mixture was put into the microwave. The microwave apparatus was previously assembled with reflux to facilitate the extraction process, as shown in Figure 3.

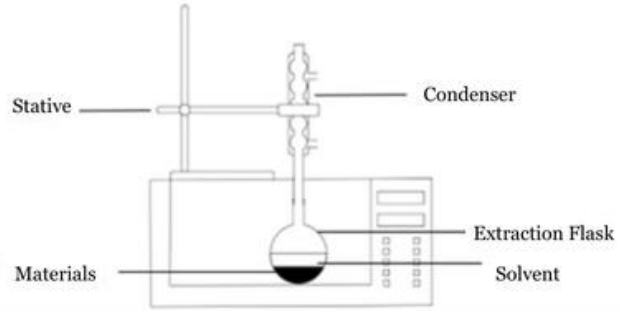

Figure 3. Microwave Extraction

This extraction uses 300 watts of microwave power. According to our previous study, high power can cause the degradation of cellulose compounds, thus reducing yield. The time variables selected are 20, 40, and 60 minutes; the selection of these variables depends on several studies from the same alkaline method literature.

The finished extraction mixture is then washed with distilled water until the pH is neutral. The following process is bleaching, which removes the lignin content in the extract (Dewatisari, 2020). The mixture was added H_2O_2 with variations 10, 20, and 30% w/w stirred for 2 hours at 125°C and 200 rpm.

Adding H_2O_2 aims to improve the yield of cellulose extracts and remove non-cellulose components such as lignin, pectin, and hemicellulose. H_2O_2 is a bleaching and oxidizing agent that helps separate cellulose from non-cellulosic materials. The bleaching results are filtered and washed with distilled water until the pH is neutral and then dried at 60°C.

Experimental Design

Some of the selected variables affect the extraction performance results. Therefore, optimization is required to determine and compare which parameters are most optimal for extraction.

After determining the upper and lower limits on the variables, the Response Surface Methodology (RSM) with BBD (Box-Behken Design) type was used. The variables considered include NaOH concentration (1, 2, and 3%), H_2O_2 concentration (10, 20, and 30%), and extraction time (20, 40, and 60 minutes). With the help of Box–Behnken statistical design, 17 runs were performed as given in Table 2.

Table 2. BBD Design Expert

Run	A:NaOH Concentration (%)	B:H_2O_2 Concentration (%)	C:Time Minutes
1	1	20	60
2	3	20	60
3	2	30	60
4	3	30	60
5	2	20	40
6	3	20	20
7	2	30	20
8	1	10	40
9	2	10	60
10	2	20	40
11	2	20	40
12	2	20	40
13	2	20	40
14	1	30	40
15	3	10	40
16	1	20	20
17	2	10	20

Cellulose Acetate Synthesis

10 g of dried cellulose was added with 24 ml glacial acetic acid and stirred at 38°C for 1 hour. After 1 hour of stirring, 40 ml glacial acetic acid and 0.08 ml sulfuric acid were added and stirred for 45 minutes. (Yulandri, 2020). The mixture was cooled to a temperature of 20°C. The resulting mixture was then added 25 ml of anhydrous acetic acid and 0.6 ml of sulfuric acid stirred again. The temperature was raised to 38°C for 1.5 hours. Adding anhydrous acetic acid aims to replace cellulose hydroxyl groups with acetyl groups (Darmawan, M. T., Elma, M., & Ihsan, 2018). The mixed solution was then added 10 ml distilled water and 20 ml glacial acetic acid and stirred again for 1 hour at 50°C. The solution was centrifuged, and a precipitate was obtained, which was then added to 500 ml of distilled water to form white cellulose acetate flakes. The resulting bath was then filtered and washed with distilled water until the pH was neutral. The results were then oven-dried at 60°C for 4 hours.

Characterization

The cellulose acetate samples were characterized using several methods: Degree of Acetylation, Fourier Transform Infrared (FT-IR), and X-ray diffraction (XRD). The degree of acetylation was analyzed using the NaOH titration method. Cellulose acetate 0.1 gram was added to 5 ml of 0.25 M NaOH and 5 ml of ethanol and allowed to stand for 24 hours. The mixture was added 10 ml of 0.25 M HCl and allowed to stand for 30 minutes. The solution was then added to 3 drops of pp indicator and titrated with 0.25 M NaOH.

$$\%AG = \frac{[(V_{bi}+V_{bt})M_b]-(V_a.M_a)}{m_{CA}} \quad (2)$$

Descriptions :

%AG = Percentage of acetyl groups
V_{bi} = Volume of NaOH added to the system (L)
V_{bt} = Volume of NaOH removed in titration (L)
M_b = NaOH Concentration (M)
V_a = Volume of HCl added to the system (L)
M_a = HCl Concentration (M)
m_{CA} = Weight of cellulose acetate sample

The measurement of acetyl content is used to calculate the Degree of Substitution (DS) with :

$$DS = \frac{162.11.AG}{43.100-(43-1)AG} \quad (3)$$

FT-IR characterization involves infrared spectroscopy to analyze the chemical structure and composition of cellulose and cellulose acetate samples. XRD analysis provides an overview of the crystalline structure of cellulose and its crystal orientation pattern.

RESULTS AND DISCUSSION

Moisture Content of Materials

Screw pine leaves were prepared and then calculated for water content. Water content is the amount of water in the material (Nuryati, Amalia, & Hairiyah, 2020). The results of the water content test of screw pine leaves can be shown in Table 3. The moisture content obtained using the oven method was an average of 7.5%. The standard for moisture content in samples is ≤10% because moisture content exceeding 10% will cause microbial growth in the sample (Utami, Umar, Syahruni, & Kadullah, 2017). This process is carried out with oven heating at temperature of 105 °C for 5 hours until the sample weight is constant. Standard deviation is a formula for measuring the distribution of a data set based on the average. The standard deviation value obtained in the

water content of screw pine is 0.033. This standard deviation value can be said to be favorable if the value is below the average water content value (Hidayat, Sabri, & Awaluddin, 2019).

Table 3. Moisture content test results

Expe-riment	Initial weight (g)	Final weight (g)	Water Content (%)	Average (%)	Standard Deviation
1	3	2.772	7.60 %		
2	3	2.774	7.53 %	7.53 %	0.033
3	3	2.773	7.56 %		

Cellulose Yields

The extraction process can affect the chemical composition of the product. Screw pine cellulose is extracted using MAE with NaOH solvent. This treatment aims to remove and hydrolysis hemicellulose, lignin, soluble mineral salts, silica, and ash (Sheltami et al., 2012). The following process is bleaching to optimally remove the lignin content in screw pine leaves (Hermayanti, Lailatul, & Amalia, 2019). The resulting cellulose is yellowish-white, as shown in Figure 4.

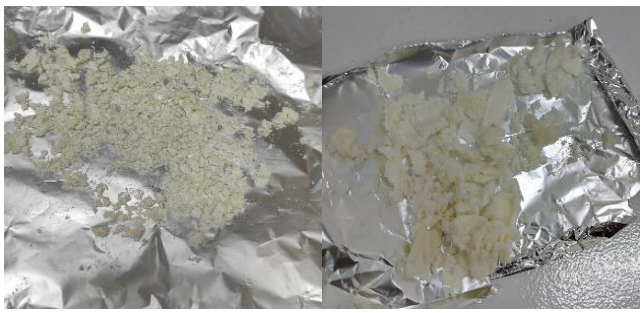

Figure 4. Extracted cellulose

Cellulose yields using the MAE (Microwave Assisted Extraction) method ranged from 45% to 51% (Table 4). Extraction with this method produced the highest yield of 50.7% in the fourth run, using 3% NaOH concentration and 30% H_2O_2 concentration with an extraction time of 60 minutes. NaOH acts as an alkaline substance in the extraction process; the higher concentrations of NaOH can increase the yield of product cellulose to some extent. As the alkali concentration increases, the cell walls break down so cellulose can be extracted more efficiently. H_2O_2 plays a role in the oxidation of cellulose fibers, helping to remove non-cellulosic substances and breaking lignin bonds. Higher concentrations can increase oxidation efficiency and reduce contamination of other substances. The 60-minute time is the most extended extraction time variable, so a longer extraction time provides enough time for NaOH to interact with cellulose.

Table 4. Yield Result

Run	Factor 1 A % NaOH	Factor 2 B % H2O2	Factor 3 C Minutes	Response Yield %
1	1	20	60	49,52
2	3	20	60	50,51
3	2	30	60	49,86
4	3	30	60	50,7
5	2	20	40	48,8
6	3	20	20	47,92
7	2	30	20	46,83
8	1	10	40	47,56
9	2	10	60	48,54
10	2	20	40	48,4
11	2	20	40	48,32
12	2	20	40	48,53
13	2	20	40	48,85
14	1	30	40	48,26
15	3	10	40	47,65
16	1	20	20	46,25
17	2	10	20	45,97

Optimization of extraction conditions

Applying the BBD (Box-Behnken Design) method in the optimization design, the data can be put as follows in Table 4. Evaluation of ANOVA is done through the lack of fit test as shown in Table 5. The results of this calculation aim to predict the response value generated by each parameter of the extraction condition, focusing on the significance value of each parameter. Based on the predetermined value, the acceptable significance value is $p<0.05$ (Setyaningsih, Saputro, Carrera, & Palma, 2019).

The lack of fit test results for the yield response using linear regression showed a p-value of 0.0001 and R^2 of 0.9211. Through the ANOVA test, the regression model for the yield response has a sufficient significance level, indicating that this model can be used to model the optimal conditions in cellulose extraction from screw pine using the MAE method.

The Predicted R^2 of 0.6813 is in reasonable agreement with the Adjusted R^2 of 0.8737 shows that the difference in this model is less than 0.2 (listed in Table 6). It could be gleaned from this result that the percentage of cellulose extracted from screw pine can best be represented by the regression model.

Analysis of the results of the 3D Surface Plot of the three variables can be seen in Figure 5. Figure 5 shows plot (a) the relationship between H_2O_2 concentration and NaOH concentration, plot (b) the

relationship between NaOH concentration with time, and plot (c) the relationship between H_2O_2 concentration with time. In plot (a), the relationship between NaOH concentration and H_2O_2 concentration was obtained, which is mutually bound, as evidenced by the increased concentration of NaOH and H_2O_2 and the greater yield produced. Plots (b) and (c) revealed that time is an influential parameter on MAE, as evidenced by the extended extraction time, the greater yield produced.

Table 5. ANOVA test result of linear regression model of yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	25.81	6	4.30	19.44	< 0.0001	Signi-ficant
A-NaOH Concen tration	1.83	1	1.83	8.26	0.0165	
B-H_2O_2 Concen tration	2.62	1	2.62	11.86	0.0063	
C-Time	16.41	1	16.41	74.17	< 0.0001	
AB	0.2695	1	0.2695	1.22	0.2956	
AC	0.2178	1	0.2178	).9844	0.3445	
BC	0.0227	1	0.0227	).1025	0.7555	
Residual	2.21	10	0.2213			
Lack of Fit	1.99	6	0.3315	5.92	0.0534	not signi ficant
Pure Error	0.2238	4	0.0559			
Cor Total	28.03	16				

Table 6. Model fit statistics on ANOVA

Std. Dev.	0.4704	R^2	0.9211
Mean	48.38	Adjusted R^2	0.8737
C.V. %	0.9723	Predicted R^2	0.6813
		Adeq Precision	15.6506

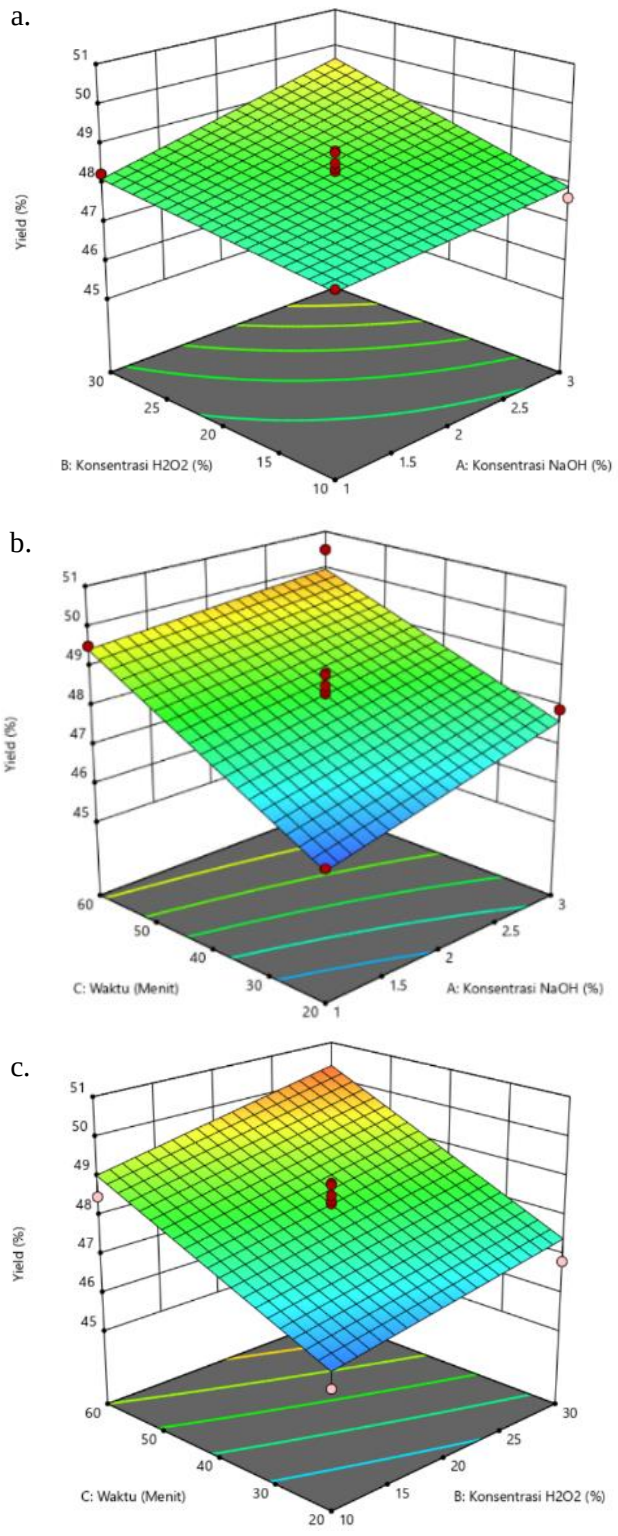

Figure 5. Response Surface Plot

Characterization of FTIR Analysis

Analysis of cellulose and cellulose acetate from screw pine was done using FTIR to identify the functional groups (Indah, Hatimah, & Hulyadi, 2021). Based on FTIR analysis of cellulose as seen in Figure 6, there is an absorption peak of the stretching

vibration of O-H at 3433 cm^{-1}. In cellulose acetate, the O-H absorption peak decreased due to the replacement with acetyl; there was an absorption of (C=O) carbonyl groups and (C-O) groups from acetyl groups at wavenumber around 1734 cm^{-1} and 1256 cm^{-1}, respectively. These peaks indicate the formation of cellulose acetate compounds. Cellulose acetate synthesis is declared successful if there is an absorption peak of (C=O) carbonyl and ester (C-O) of acetyl groups (Asparingga, Syahbanu, & Hairil Alimuddin, 2018).

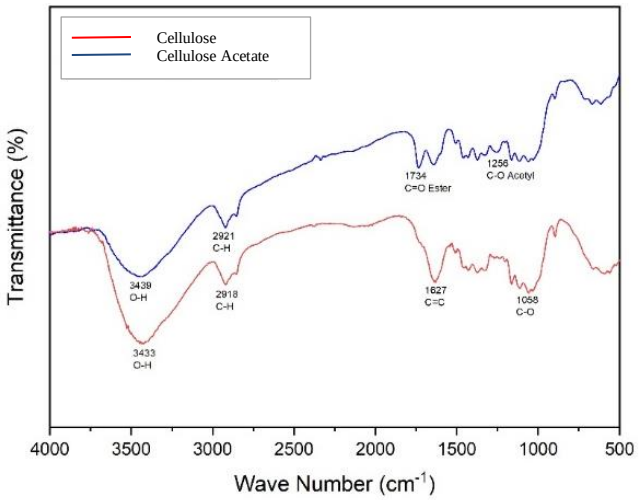

Figure 6. FTIR Spectra of cellulose and cellulose acetate

Characterization of XRD Analysis

Analysis of cellulose from screw pine using the X-Ray Diffraction (XRD) aims to identify the crystal structure. The basic principle of the XRD method involves firing a monochromatic beam at a sample, which causes reflection and scattering of the beam in all directions. When interference occurs in only one phase, the result is a reflection focused in a specific direction. This combination of reflection and interference produces a diffraction pattern, reflecting the cellulose's crystalline structure (Asparingga et al., 2018). Figure 7 shows the sample's X-ray diffraction (XRD) characterization results.

In Figure 7, the diffraction peak around 22.53798° shows cellulose has a crystalline region. The higher peak intensity indicates a higher degree of crystallinity, which differs from the amorphous characteristics (Nurlia, Anas, & Erniwati, 2020). Typical diffraction peaks for cellulose appear in the 2θ range around 15° and 22° (Julie Chandra, George, & Narayanankutty, 2016).

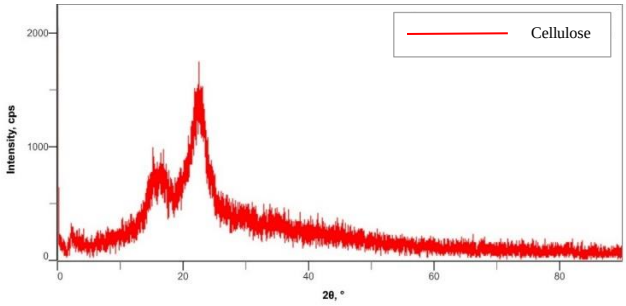

Figure 7. X-ray diffractogram of cellulose

Acetylation Degree Analysis of Cellulose Acetate

The resulting cellulose acetate has a Degree of Substitution (DS) of 2.045 and a percentage of acetyl groups of 35.475%, which shows characteristics as triacetate. High lignin solubility is crucial in the extraction process to achieve high DS. Lignin competes with cellulose in the acetylation reaction, so high lignin content can reduce the yield at the acetylation stage (Utami et al., 2017).

In this case, the relatively high DS, more than 2, indicates that most of the hydroxyl groups on the cellulose had been replaced by acetyl groups. In addition, the percentage of acetyl groups indicates that about 35.475% of the cellulose mass has been substituted with acetyl groups during the acetylation process. Overall, the results of this analysis indicated that the cellulose acetate synthesis achieved a high degree of substitution and gave a product with a significant proportion of acetyl groups. These properties could have implications on the physical and chemical properties of cellulose acetate, such as its solubility, strength, and reactivity in various applications such as filtration processes, membranes, biofilms, plastics, sensors, and coating materials (Souhoka & Latupeirissa, 2018).

CONCLUSION

Screw pine leaves are a biomass that contains cellulose with limited utilization. The highest cellulose yield obtained in the extraction process is 50.7% with an operating condition of 3% NaOH, 30% H_2O_2, and 60 minutes. XRD analysis shows the diffraction peak of 2θ = 22.53798° The structure of screw pine cellulose is almost similar to commercial cellulose. The cellulose produced from screw pine was reacted to form cellulose acetate. FTIR identification of cellulose acetate showed the presence of carbonyl (C=O) and (C-O Acetyl) group absorption at wavenumbers 1734 cm^{-1} and 1256 cm^{-1}, respectively. The acetyl content of cellulose acetate from screw pine produced is 35.475%. In summary, this study provides information about the potential of

screw pine leaves as raw material for cellulose acetate fabrication.

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