

Comparison of Cellulose Extraction Methods from Lontar (*Borassus flabellifer*) and Salak (*Salacca zalaacca*) Fronds

Aisyah Rusdin^{1,*}, Rahmah Harun², Vivi Alfi Yunita³, Awaluddin⁴

¹Department of Chemistry, Sipatokkong Mambo University, Bone, Indonesia

^{2,3}Department of Chemistry, Alauddin State Islamic University, Makassar, Gowa, Indonesia

⁴Department of Management, Syekh Yusuf Al-Makassari University, Gowa, Indonesia

*e-mail correspondence: aisyahrusdin1@gmail.com

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ABSTRACT

Palmyra fronds and salak fronds are waste products from the palmyra and salak plants that have not been optimally utilized. However, the high cellulose content in these fronds offers potential applications across various fields. This study aimed to extract and compare cellulose from lontar (*Borassus flabellifer*) and salak (*Salacca zalaacca*) frond waste using two methods: alkaline sodium hydroxide (NaOH) solvent and nitric acid (HNO₃) hydrolysis. After soaking and heating, the extraction was performed through a bleaching process. The yield results showed that lontar fronds produced the highest yield of 64.22% using the acid hydrolysis method, while salak fronds yielded 46.8%. The cellulose obtained from lontar fronds was gray, and from salak fronds, it was white, indicating differences in purity. After treatment, the disappearance of the carbonyl group (C=O) in the FTIR functional group analysis indicated successful delignification. Common cellulose functional groups such as O-H, C-H, and C-O, as well as β -1,4 glycosidic bands, were detected at wave numbers 895-897 cm⁻¹, indicating that the cellulose structure was well preserved. Cellulose from both lontar and salak fronds has great potential to serve as an environmentally friendly alternative raw material for applications in bioplastics, bioethanol, and other cellulose derivatives.

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INTRODUCTION

Indonesia is the world's largest tropical archipelagic country [1]. The country is home to a diverse range of flora and fauna. Therefore, to enhance the value of local natural resources, efforts are required to utilize them optimally [2]. Sorghum stalks [3], lontar fronds, and salak fronds are waste materials that are underutilized by society. Lontar (*Borassus flabellifer* Linn.) is a type of palm or Arecaceae that primarily grows in dry regions. Lontar is found from Saudi Arabia to Indonesia, and it is abundant in the provinces of East Nusa Tenggara, East Java, and South Sulawesi in Indonesia [4]. The chemical composition of lontar fronds includes 54.27% cellulose, 22.34% hemicellulose, and 1.12% lignin [5].

Salak fronds (*Salacca zalaacca* (Gaertner) Voss) are plants containing carbohydrates and are available in large quantities, but are not widely utilized [6]. The chemical composition of salak fronds includes 31.7% cellulose, 33.9% hemicellulose, and 17.4% lignin [7]. Therefore, optimal utilization of lontar and salak frond waste can be achieved through cellulose extraction.

Cellulose ($C_6H_{10}O_5$)_n is a primary material for producing biomaterials such as bioplastics [8], cosmetics [9], food products [10], and other medical materials due to its biocompatibility, biodegradability, and non-toxicity [11]. The cellulose extraction process can be carried out using enzymatic hydrolysis [12], acid hydrolysis [13], and alkaline solvents [14]. Enzymatic hydrolysis for cellulose production requires a long processing time of approximately 48, 72, and 96 hours. Hydrolysis using concentrated acids such as nitric acid (HNO_3) [15] and hydrochloric acid (HCl) can accelerate the reaction [16]. A study [17] conducted cellulose extraction using sulfuric acid (H_2SO_4) with raw materials such as ramie fibers and kraft pulp [18]. Another study [19] also performed cellulose extraction from coconut belt fibers using peracetic acid, yielding cellulose at 83.14%.

Cellulose extraction using alkaline solvents, specifically sodium hydroxide ($NaOH$) [20], [21], is a well-established method. The extraction process with $NaOH$ as a cooking solution is adjusted with a suitable ratio and applied under high pressure [22]. This method is more efficient in removing lignin and hemicellulose, resulting in purer cellulose in a relatively short period. The use of alkaline solvents also offers advantages in terms of lower cost and the absence of hazardous waste. Therefore, the alkaline solvent extraction method and acid hydrolysis are selected in this study due to their advantages in efficiency, speed, and more environmentally friendly waste processing. This is supported by a study [23] using an alkaline solvent with sugarcane bagasse as raw material, yielding 85.95% cellulose.

This article reports a comparison of cellulose extraction methods from lontar fronds and salak fronds using alkaline solvents and acid hydrolysis. The study provides an innovative approach to developing cellulose from agricultural waste, specifically lontar and salak fronds, which can be used as raw materials for producing biodegradable bioplastics.

The novelty of this research lies in its contribution to the field of cellulose extraction from agricultural waste, particularly lontar and salak fronds, which have been underutilized. This study compares two different cellulose extraction methods—alkaline solvents and acid hydrolysis—for the first time on these two types of waste. While both extraction methods are well-documented in the literature, there has been limited comparison of these methods on lontar and salak fronds, making this study a new perspective on the effectiveness and efficiency of each method in producing high-quality cellulose. Another innovation is the focus on utilizing abundant agricultural waste in Indonesia for cellulose extraction, which not only adds economic value but also offers a solution to reduce agricultural waste. By comparing these extraction methods, the study provides a foundation for further development in using cellulose as a raw material for bioplastics or other applications in the future.

MATERIALS AND METHODS

Materials

This study uses distilled water, 3.5% HNO_3 , 10% H_2O_2 , 2% sodium hydroxide ($NaOH$), lontar fronds, and salak fronds. The equipment used in this study includes a Fourier Transform Infrared (FTIR) spectrometer, hotplate stirrer, analytical balance, 100 mesh sieve, oven, electric stove, and glassware.

Procedures

a. Sample Preparation

The samples used are lontar and salak fronds. The samples are obtained from Wajo District, Maniangpajo Subdistrict, Anabanua Village, South Sulawesi. The samples are washed and dried under sunlight. After drying, the samples are ground using a grinding machine. The ground samples are then sieved using a 100 mesh sieve [23].

b. Cellulose Extraction Using Acid Hydrolysis

A total of 75 grams of lontar frond powder is added to 10 mg NaNO_2 and 2 L of 3.5% HNO_3 . The mixture is then heated for 2 hours with stirring at 90°C using a hotplate stirrer. After heating, filtration is performed, and the residue is washed with distilled water until neutral. The residue is then treated with 375 mL of 2% NaOH and 375 mL of 2% $\text{Na}_2\text{S}_2\text{O}_3$ and heated for 1 hour at 50°C . Filtration is repeated, and the residue is neutralized using distilled water. The same procedure is performed using salak fronds [24].

c. Cellulose Extraction Using Sodium Hydroxide (NaOH)

A total of 50 grams of lontar frond powder is added to 500 mL of 10% NaOH . The mixture is stirred thoroughly and left to soak for 24 hours. Then, filtration is carried out using a filter cloth to obtain cellulose residue. The same process is performed using salak fronds [23].

d. Bleaching Process

The cellulose residue obtained from the extraction is mixed with 500 mL of 1.75% NaOCl solution and heated for 30 minutes at 70°C . Filtration is carried out, and the residue is neutralized. Then, 500 mL of 17.5% NaOH is added to the solution, and it is heated at 80°C for 30 minutes while stirring on a hot plate. Filtration is repeated and neutralized. Subsequently, the residue is mixed with 250 mL of 10% H_2O_2 and heated for 15 minutes at 60°C using the hot plate. Filtration and neutralization are again performed. The residue is then dried in an oven at 60°C and stored in a desiccator [25]. Finally, cellulose yield and functional group analysis are conducted using FTIR. Cellulose yield is determined using Eq. (1):

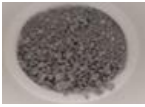
$$\text{Cellulose endumen (\%)} = \frac{\text{Dry weight of sample extract}}{\text{Weight of sample powder of sample stem}} \times 100\% \quad (1)$$

RESULTS AND DISCUSSION

Extraction is the process of separating components from a mixture using a solvent [26]. Cellulose is a glucose monomer with long polymer chains. Cellulose is a common substance in nature as it constitutes the majority of plant cell walls. Most cellulose is found in combination with lignin and hemicellulose in the cells of woody plants, but a portion can be found in its pure form, as seen in lontar and salak fronds, as well as other plants. To obtain cellulose, several methods have been used, such as alkaline treatment through bleaching processes, steam exposing, irradiation, extraction, biodelignification, and enzymatic processes. Cellulose can be extracted through these processes [27]. Lontar and salak fronds are underutilized waste materials in society that contain high cellulose content [4], [5], [7]. This study extracts cellulose from lontar frond powder and salak frond powder using NaOH solution and HNO_3 solution.

The results of cellulose extraction are shown in Table 1. In Table 1, the cellulose from lontar fronds produced in this study is gray, while the cellulose from salak fronds is white. The color difference is due to variations in the sample and lignin content [5]. The cellulose yield was calculated by comparing the dry weight of cellulose to the amount of sample powder used. The total cellulose yield obtained from lontar fronds using alkaline solvent and acid hydrolysis were 64.22% and 11.06%, respectively. Meanwhile, the cellulose yield obtained from salak fronds using alkaline solvent and acid hydrolysis were 46.8% and 17.2%, respectively.

Table 1. Cellulose Extraction Methods from Lontar and Salak Fronds

Sample	Extraction	Initial weight (g)	Final weight (g)	Yield (%)	Color	Image
Palm fronds	Alkali	75 g	8.3	11.06	Abu	
	Acid hydrolysis	50	32.1	64.2	Abu	
Salak fronds	Alkali	75	12.9	17.2	Putih	
	Acid hydrolysis	50	23.4	46.8	Putih	

Based on the cellulose yield obtained, it can be concluded that lontar fronds and salak fronds have the potential to be further developed in cellulose discovery, making them suitable as raw materials for bioplastic production.

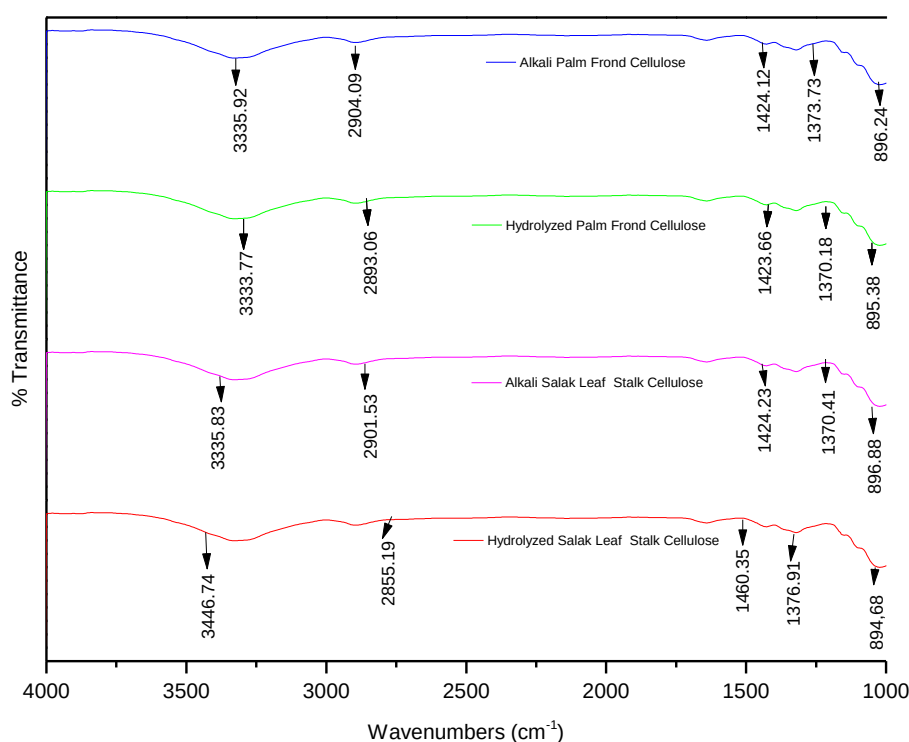


Figure 1. FTIR Spectrum of Cellulose from Lontar Fronds and Salak Fronds

Table 2. FTIR wave numbers of lontar frond powder, cellulose from lontar fronds, using an alkaline method, and cellulose from lontar fronds using the acid hydrolysis method

Absorption Area (cm ⁻¹)				
Palm powder	leaf	Palm leaf cellulose alkali method	Palm leaf cellulose acid hydrolysis method	Functional groups
3426.1		3335.92	3333.77	O-H
2928.8		2904.09	2893.06	C-H
1735.7		-	-	C=O
1510.3		-	-	C=C
1425		1424.12	1423.66	C-H
1378.6		1370.73	1370.18	C-H
1249.4		1264.75	1200.57	C-O-C
-		1159.89	1158.04	C-O
897.6		896.24	895.38	C-H

Table 3. FTIR wave numbers of salak frond powder, cellulose from salak fronds using an alkaline method, and cellulose from salak fronds using the acid hydrolysis method

Absorption Area (cm ⁻¹)				
Salak leaf powder	Salak leaf	Salak leaf cellulose alkali method	Salak leaf cellulose acid hydrolysis method	Functional groups
3340.7		3335.83	3446.74	O-H
2917.6		2901.53	2855.19	C-H
1730.6		-	-	C=O
1504.5		1540.43	-	C=C
1421.5		1424.23	1460.35	C-H
1370.6		1370.41	1376.91	C-H
1236		1201.41	1230.52	C-O-C
-		1159.53	1162.08	C-O
897.02		896.88	894.68	C-H

Figure 1, Table 2, and Table 3 show the FTIR spectra of lontar frond powder and salak frond powder, cellulose from lontar fronds and salak fronds using the alkaline method and acid hydrolysis method. In Table 2, before the lontar frond powder and salak frond powder were treated, functional group analysis was performed using FTIR, which resulted in peaks at wave numbers 1735.7 and 1730.6 for the C=O group. This indicates that the lontar and salak frond powders before treatment still contain lignin. This is consistent with the study [28] that the region at 1702 cm⁻¹ shows the presence of the carboxylate C=O group of lignin. From the results of the study, after treatment to form cellulose, the wave numbers 1735.7 and 1730.6 cm⁻¹ disappeared, indicating the partial removal of lignin compounds from the lontar and salak frond powders. The functional groups of the FTIR spectra of cellulose from lontar fronds using acid hydrolysis and alkaline solvent methods each showed the presence of the O-H group at wave numbers 3333.77 and 3335.92 cm⁻¹, the C-H group at wave numbers 2893.06 cm⁻¹ and 2904.09 cm⁻¹, and the C-O group at wave numbers 1158.04 cm⁻¹ and 1159.89 cm⁻¹.

The functional groups in the FTIR spectra of cellulose from salak fronds using acid hydrolysis and alkaline solvent methods each showed the presence of the O-H group at wave numbers 3446.74 cm⁻¹ and 3335.83 cm⁻¹, the C-H group at wave numbers 2855.19 cm⁻¹ and 2901.53 cm⁻¹, and the C-O group at wave numbers 1162.08 cm⁻¹ and 1159.53 cm⁻¹. The presence of the β -1,4 glycosidic group and C-O-C stretching vibration in cellulose from lontar fronds using acid

hydrolysis and alkaline solvent methods was indicated by peaks at 895.38 cm^{-1} and 896.24 cm^{-1} , respectively. Meanwhile, the presence of the β -1,4 glycosidic group and C-O-C stretching vibration in cellulose from salak fronds using acid hydrolysis and alkaline solvent methods was indicated by peaks at 894.68 cm^{-1} and 896.88 cm^{-1} . This demonstrates the specificity of the FTIR spectrum for cellulose functional groups [29]–[32]. This is in agreement with the study [23], which found that the primary functional groups in cellulose from sugarcane bagasse are hydroxyl ($-\text{OH}$) and C-H groups. Therefore, cellulose has the potential to be used in various applications such as bioplastics [33], bioethanol [34], carboxymethyl cellulose [35], and others.

CONCLUSION

This article reports that both acid hydrolysis and alkaline treatment methods successfully extracted cellulose from lontar frond and salak frond waste. The extraction results show that the cellulose yield from lontar fronds is higher compared to salak fronds, with the acid hydrolysis method yielding the highest value of 64.22%. The difference in color of the cellulose extracted from both types of fronds indicates a variation in the lignocellulose content. FTIR analysis confirms the success of the extraction process, evidenced by the appearance of characteristic cellulose groups such as O-H, C-H, and C-O, as well as the disappearance of the carbonyl group ($\text{C}=\text{O}$), indicating the removal of lignin. The presence of the β -1,4 glycosidic group, indicated by peaks in the $895\text{--}897\text{ cm}^{-1}$ region, shows that the basic cellulose structure has been well preserved. Therefore, cellulose extracted from salak fronds and lontar fronds has great potential to be used as an alternative raw material for various applications, including bioethanol, bioplastics, and others.

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