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Short Communication

Isolation and Characterization of Cellulose from Siwalan (*Borassus flabellifer*) Fruit Peel Fiber

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ABSTRACT

Borassus flabelliger's fruit peel fiber is one of the plantation wastes that has not been optimally used. Meanwhile, it contains several crude fibers including cellulose, which is sufficient and can be isolated for various purposes. The aim of this study was to determine the characteristics of cellulose from siwalan fruit peel fiber (*Borassus flabellifer*). The isolation was conducted through a chemical process that comprises hydrolysis, delignification, and bleaching. Furthermore, water content, pH, form and Fourier Transform-Infrared (FTIR) analysis were the physical characterization and standardization parameters used to assess the *Borassus flabellifer* cellulose. The tests carried out showed that the isolate had a pH of 6.8 and a moisture content of 7.34%. The Fourier Transform-Infrared (FTIR) showed absorption at 3323.34 for O-H, 2893.22 for C-H, 1371.38 for C-O-H, and 1157.28 for C-O, which revealed that the values are the typical cellulose peaks. The results showed that cellulose isolated from *Borassus flabellifer* fruit peel fibers has numerous similarities with the standard synthetic cellulose used. Additionally, a large cellulose yield of 12.3% (w/w) was obtained from the fruit peel, hence, further development is needed because the isolate can serve as an alternative for animal gelatin made from pigskin. It is also a halal guaranteed product and can fully meet various pharmaceutical industries' needs.

Keywords: *Borassus flabellifer*, Siwalan, Isolation, Cellulose, Characterization.

Introduction

Indonesia has the largest biodiversity in the world, and this has great potential benefits for the country. Therefore, efforts are needed to develop ways on how to convert the country's natural resources into more valuable materials, such as veterinary foods, wood, paper, fibers, clothes, cosmetics, and pharmaceutical products.[1] Siwalan (*Borrasus flabellifer*) is a typical Indonesian flora that grows in dry climates surrounding the country, such as in East Java (Tuban, Madura), NTT (Sumbawa, Timor, Rote, Sabu), South Sulawesi, and Southeast Maluku.[2] Subsequently, the Semanding district in Tuban City has the highest percentage of the plant, but its plantation wastes have not been fully utilized.[2] The waste is only being used as food ingredient due to its high nutritional value, but after being processed, it is often disposed of as a low economic value waste. Meanwhile, it contains 68.94% cellulose, 5.37% lignin, 14.03% hemicellulose, and 0.6% wax.[3] Siwalan fruit peel waste causes environmental problems when stored in a place. However, environmental hazard is a normal phenomenon that occurs globally, and cannot be separated from human life. It is often trigged by humans' desire to take advantage of nature as well as the improper use/disposal of waste. Therefore, siwalan waste's utilization can be optimized by developing research on the production of cellulose from siwalan fruit peel. Cellulose ($(C_6H_{10}O_5)n$) is one of the essential raw materials for manufacturing pharmaceutical products, cosmetics, bioplastics, and food packaging materials. It is often found in plants as a cell wall-forming material.

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Furthermore, it is a natural polymer with a linear structure, crystalline shape, but it is not readily soluble. Cellulose is a polysaccharide containing a polymer of glucose units that are bonded together by the 1,4-glycosidic bond to form a long and straight-chain.[4]
There are several ways or methods used to isolate cellulose, such as mechanical methods: ultrasonic and high pressure; chemical methods: strong acid hydrolysis, organoleptic, alkaline solvent, oxidation, and ionic liquids; and biological method using enzyme. Meanwhile, the method used in this study was the chemical cellulose isolation process.[5] The process began with hydrolysis using solid acids, such as sulfuric acid, hydrochloric acid, and perchloric acid. Strong acids efficiently hydrolyze the amorphous part of extended plant fiber to become shorter.[6] They also help to hydrolyze hemicellulose into xylose and other sugars. Furthermore, the second stage involves the removal of lignin or delignification using NaOH solution, while the third stage is a bleaching process used to facilitate the degradation of lignin and other impurities that have not been lost during the chemical process. Oxidizing chemical compounds, such as hypochlorite and hydrogen peroxide are usually used in the bleaching process. Subsequently, the cellulose obtained was characterized using FTIR, while the synthesis success interpretation was carried out by comparing the isolate gotten from the siwalan fruit peel to synthetic cellulose (Sigma-Aldrich®). The aim of this study was to determine the characteristics of cellulose from siwalan fruit peel fiber (*Borassus flabellifer*) in the process of making cellulose. This study may open up an avenue for the development of cellulose from siwalan fruit peel, which can serve as an alternative to animal gelatin made from pigskin.

Materials and Methods

Materials

Siwalan (*Borrasus flabellifer*) fruit peel fibers powder was prepared from Siwalan fruit collected from Semanding district, Tuban city, East Java, Indonesia. All the chemicals used including HNO_3, NaOH, H_2O_2 were purchased from Merck. Meanwhile, the standard cellulose used was the Sigma-Aldrich synthetic cellulose.

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The samples were characterized by their physical properties and by FTIR spectrophotometer (Shimadzu FTIR 8400S). They were then analyzed using a KBr plate in the 4,000-400 cm^{-1} range. Other materials used for the study include a set of lab-wares, 100 mesh, and 120 mesh sieved, analytical balance (Ohaus Pioneer PA214), hot plate, magnetic stirrer (IKA), oven (Binder), blender (Philips), mixer, sonicator, pH meter (Senz pH), and moisture content analyzer (HC103).

Preparation of sample

The Siwalan (*Borrasus flabellifer*) fruit peel fibers used as a sample for glucose isolation were obtained from the Semanding sub-district of Tuban City. Subsequently, the samples were authenticated at the Materia Medica Laboratories, Batu City, Indonesia. The fiber was obtained by peeling the fruit, the outer skin was then removed and washed with water. The fibers were cut (~ 1 mm), washed in distilled waters, and then dried for 24 h at 50^{o}C. The dried samples were then converted to powder with a water content specification of approximately 7% and a particle size of 250-micron

Isolation of cellulose

Cellulose is physically and chemically present in nature, where it is surrounded by hemicellulose and lignin to form a lignocellulose matrix.[7] Cellulose was isolated from siwalan (*Borrasus flabellifer*) fruit peel fibers waste by chemical method. The siwalan fruit powder (50 g) was hydrolyzed using 400 mL of 4% HNO_3 at 80°C for 2 hours, and then filtered and bleached using 200 mL of 10% H_2O_2 at 80°C for 1 hour. Subsequently, the delignification process was carried out using 200 mL of 2N NaOH at 80°C for 1 hour, then filtered and bleached once using 200 mL of 10% H_2O_2 at 80°C for 1 hour. The cellulose obtained was then oven-dried at 70°C for 24 h.[8]

Characterization of cellulose from Siwalan (Borrasus flabellifer) compared with standard cellulose

Physical characterization

Basic characterization testing was used to ensure the success of the siwalan fruit peel synthesis. Furthermore, experiments carried out include physical tests, namely organoleptic and moisture content analysis as well as chemical tests for pH, and functional group using FTIR characterization. The synthesis results were shown by the similar characterization results between the cellulose obtained from the siwalan fruit peel fiber and synthetic cellulose (Sigma-Aldrich®).

Fourier-Transform Infrared Spectrometer (FTIR)

The FTIR spectroscopy was used to determine the functional groups of siwalan (*Borrasus flabellifer*) fiber, siwalan cellulose, and standard cellulose. It was also used to identify the functional groups changes that occurred while converting the waste to cellulose.[3-6] The FTIR analysis was carried out using an FTIR spectrophotometer FTIR 8400S (Shimadzu, Kyoto, Japan), and the spectra results of siwalan fibers, siwalan cellulose, and standard cellulose were then compared with each other.

Results and Discussion

Isolation is the process of separating natural compounds using an appropriate solvent. Cellulose is a material found in plant cell walls, woody materials, seed hair, tree bark, and marine plants. Meanwhile, siwalan fiber is one of the plants with a high cellulose content.

This study isolated cellulose from siwalan fruit powder using a solution containing 4% HNO_3, 2N NaOH, and 10% H_2O_2. Yield calculation is very essential because it is used to determine the amount of cellulose obtained during the synthesis. The calculation can be performed by finding the ratio between the cellulose dry weight and the amount of siwalan fruit peel used. Based on the yields obtained, it can be seen that siwalan has the potential to be further developed in the discovery of cellulose so that it can be used as a raw material for halal drugs. The total yield obtained was 12.3% from 20 parts (w/w). From an economic point of view, this number is very good, although not perfect. This research is an initial research in the development of cellulose from siwalan so that further development can be carried out in its synthesis methodology which can produce more yield.

The hydrolysis step in the isolation process is used to release hemicellulose from the cellulose structure. Hemicellulose has shorter chains than cellulose chains, and its polymers do not have a straight shape. However, it has branched polymers with a non-crystalline form, which makes it easier to dissolve in solvents and react with solutions than cellulose.[12] The delignification stage was carried out using NaOH solution that can damage the lignin structure in the crystalline and amorphous parts, thereby preventing the separation of lignin. Meanwhile, the bleaching process was used to dissolve the remaining lignin compounds that can cause color changes by degrading short-chain lignin chains. This degradation causes the dissolution of lignin in water or alkali while washing.[13]

Physical characterization

The identification test showed that cellulose from siwalan has a pH of 6.8, while the standard cellulose had a pH of 6.3 (Table 1). Furthermore, siwalan cellulose has a moisture content of 7.34%, while standard cellulose had a value of 7.21%. These results show that they have similar physical properties. This was then reinforced with the organoleptic test, which revealed that the siwalan and standard cellulose are in the form of a white powder.

The results of physical characterization showed that the white powder form of siwalan cellulose was similar to that of standard cellulose, which was also listed in the standard cellulose CoA. The results also revealed that siwalan cellulose has a pH of 6.8, while a similar value of 6.3 was obtained from the standard cellulose. Furthermore, the water content of siwalan cellulose was 7.34%, while the standard cellulose had a similar value of 7.21%. Figure 2 and Table 2 show the isolation, characterization, FTIR spectra of siwalan (*Borrasus flabellifer*) fruit peel fiber, Siwalan cellulose, and cellulose standards. The spectroscopic examination was used to determine the functional groups of cellulose after removing hemicellulose and lignin. Figure 2 shows the vibration band of the 3 test samples.

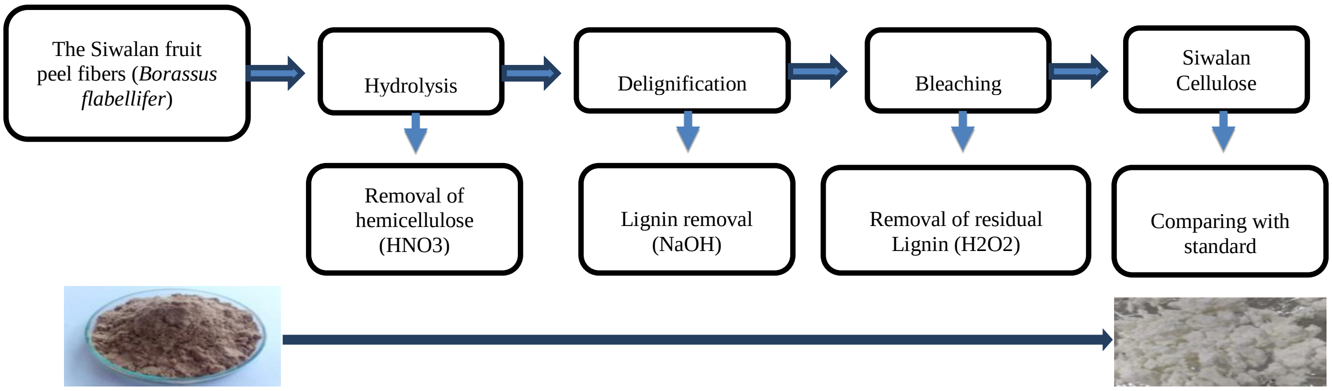

Figure 1: Schematic of the Isolation and Characterization of Cellulose from Siwalan (*Borassus flabellifer*) Fruit Peel Fiber

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The results of FTIR spectroscopy for siwalan cellulose and standard cellulose were similar, which indicates that they have similar functional groups.

Fourier-Transform Infrared Spectrometer (FTIR)

Figure 2 and Table 2 show the FTIR spectra of standard cellulose, siwalan powder, and cellulose. Hydroxyl peaks were observed at a wavenumber of 3,322 cm^{-1} for standard cellulose and 3,336 cm^{-1} for the Siwalan cellulose. -CH_2 vibrations group appeared at 2893 cm^{-1} for standard cellulose and 2,900 cm^{-1} for Siwalan cellulose. Meanwhile, the C-O vibration group was observed at 1,041 cm^{-1} for standard cellulose and 1,039 cm^{-1} for Siwalan. The C-O-H bending group that is linked with the carbon chain appeared in cellulose and siwalan cellulose at wave numbers of 1,371 cm^{-1} and 1,365 cm^{-1}, respectively.[9-14] The FTIR spectrum of the isolated cellulose also showed typical cellulose absorption bands. The absorption bands were observed at wavenumber of 1,637 cm^{-1} (siwalan cellulose) and 1,654 cm^{-1} (standard cellulose), which indicated the presence of the C-O group. Meanwhile, the band that appeared at 898 cm^{-1} indicates the presence of C-O-C stretching vibration and -1,4-glycosidic group. These results indicate a specific FTIR spectrum for the cellulose group.[14] The data in Figure 2 and Table 2 proved that siwalan cellulose and standard cellulose have the same absorption band at various wavelengths, and they also have a similar functional group. The wavenumber of siwalan fruit peel is identical to that of the standard spectrum. Furthermore, the change in functional groups indicates that the synthesis process of cellulose from the siwalan fruit peel fiber was successfully carried out.

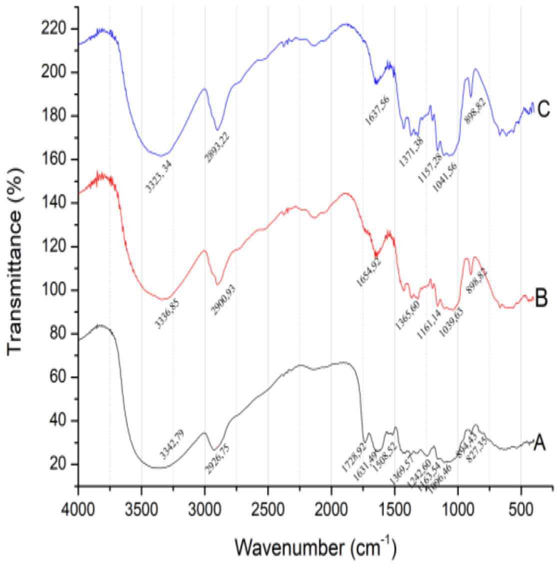

Figure 2: FTIR spectra (A) Siwalan fruit peel fiber, (B) Siwalan cellulose and (C) Standard cellulose

Table 1: Physical characterization of the cellulose from Siwalan and cellulose standard

Sample	Organoleptic		pH	Moisture content (%)
	Dose Form	Color		
Cellulose Siwalan	Powder	White	6.8 ± 0.02	7.34 ± 0.03
Cellulose Standard	Powder	White	6.3 ± 0.01	7.21 ± 0.05

All values as mean ± standard deviation (n = 3)

Table 2: Analysis of functional groups of cellulose from Siwalan and cellulose standard

Wavenumber (cm^{-1})			Range (cm^{-1})	FunctionalGroup Interpretation
Siwalan Fiber	**Cellulose Siwalan**	**Cellulose Standard**		
3342,79	3336,85	3323,34	3400-3200	*O-H stretching vibration*[9]
2926,75	2900,93	2893,22	3000-2850	*Sp^3 C-H stretching*[9]
1728,92	-	-	1750-1735	*C=O stretching*[10]
-	1654,92	1637,56	1650-1630	*C-O*[9]
1631,49 and 1440,64	-	-	1600-1475	*C=C aromatic aromatic ring*[10]
1508,52	-	-	1600-1500	*C=C stretching aromatic ring*[11]
1369,57	1365,60	1371,38	1440-1000	*C-O-H bending*[9]
1242,60	-	-	1300-1000	*C-O stretching vibration*[10]
1163,54 and 1096,46	1161,14 and 1039,63	1157,28 and 1041,56	1300-1000	*C-O stretching vibration*[9]
894,43	898,82	898,82	around 850	*Asymmetric C-O-C stretching vibration*[9]
827,35	-	-	900-690	*=C-H stretching aromatic ring*[9]

Conclusion

Based on the results obtained, it can be concluded that the cellulose characteristics of the siwalan fruit peel fiber (*Borassus flabellifer*) in the cellulose manufacturing process have the physical characteristics and the FTIR vibrations are similar to standard cellulose. Furthermore, this study revealed the potential of siwalan cellulose, which can be used as a pharmaceutical raw material. It contains a large amount of methylcellulose, ethylcellulose, carboxymethyl cellulose, hydroxypropyl methyl cellulose, hence, siwalan fruit peel fiber waste can be used as an alternative source of cellulose.

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Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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