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Synthesis of Cellulose from Decorticated Sisal Plants *(Agave sisalana)* using the Acid Hydrolysis Method

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Abstract: Sisal Plant Production Process *(Agave sisalana)* produces waste of around 95%, which is wasted and can be an environmental problem because it is not processed properly. Sisal decortication waste contains active biochemical compounds, one of which is cellulose, which has the potential to be used in various fields. Cellulose is one of the most widely distributed and abundant biopolymers on Earth, as the main source of renewable materials obtained from plant fibers. Initial Treatment of Fiber Alkalization using 5% NaOH solution (1:20) for 2 hours at a temperature of 80 °C at a speed of 200 rpm. Then the bleaching process*(bleaching)*. Samples of the results of alkalization treatment using hydrogen peroxide solution (H2THE23%) at a temperature of 80 °C for 3 hours, repeated once. In the Acid Hydrolysis process, the resulting sample is bleached with acid using sulfuric acid (H2SO465%) at a temperature of 80 °C for 1 hour (1:20). Sample Characterization Fiber characterization using the NDF test to determine cellulose content. The results of the cellulose content test in sisal fiber decortication waste were 1.545 mg/L Based on the results of the study, nanocellulose with a high % crystallinity was successfully extracted from sisal fiber decortication waste using a chemical treatment method. The FTIR spectrum shows a broad band at 3358-3410 cm^{-1}which is the vibration of the OH group of cellulose. The removal of lignin levels was successfully carried out, showing that the peak of the spectrum band produced was only 1279.26 cm^{-1}. The average size of nanocellulose particles is around 10-30 nm and consists of 30-100 cellulose molecules.

Keywords: Acid Hydrolysis; Alkalization; Decortylation; Nanocellulose; Sisal.

Introduction

Sisal fiber (*Agave sisalana*) is one of the most widely used natural fiber and is very easy to cultivate. In general, countries such as East Africa, Brazil, Thailand, and Indonesia are the main producers of sisal fiber in the world [1]. According to the Food and Agriculture Organisation, almost 4.5 million tons of sisal fiber are produced annually worldwide. Indonesia and Thailand are two Southeast Asian countries that also produce sisal [2]. Indonesia produces 500 tons of sisal fiber per year. In 2017, the Sweetener and Fiber Crops Research Institute (Balittas) as a companion, PT. Sumbawa Bangkit Sejahtera succeeded in producing sisal varieties introduced from China with a potential dry fiber production of 4,728 - 5,964.763 kg/ha/year, fiber yield of 4-5.298%, shiny yellowish white fiber with a strength of 31.363 ± 1.849 g/tex, has a one-cycle plant age of 8-13 years with a first harvest age of 36-48 months after planting. Indonesia produces 500 tons of sisal fiber/year [3].

Sisal plant *(Agave sisalana)* with the characteristics of strong leaf fibers, not stretchy and resistant to seawater. The production of dry fiber of sisal plants of the sisalana type in the world fluctuated between 2012 and 2017, with an average import of 280,550 tons and an average export of 239,150 tons [4]. The yield of sisal fiber is only 3-5% of the weight of sisal leaf biomass; the rest becomes waste. In the sisal processing industry, waste from decortication reaches 95-97%. The biomass of sisal waste that is piled up continuously without proper handling can become a problem for the environment. In the remediation separation process, waste is produced, which is disposed of in water and soil in large quantities, causing environmental pollution and a slum environment [5].

Sisal decortication waste contains active biochemical compounds, one of which is cellulose, which has the potential to be used in various fields. Sisal decortication waste is used as a woven material, bags, ropes (ropes that are resistant to sea water), carpets [6] table mats, bed mats, baskets, bowls/plates, wall clocks, jewelry [7] and can be used as raw materials for new, environmentally friendly and cheap insecticides [8]. In the automotive industry, such as car dashboards and other automotive products, raw materials for making high-quality paper/money paper, raw materials for the packaging industry and filter materials and used as paper pulp materials [9]. In addition, in the food and beverage sector, sisal waste contains stabilizer/emulsifier compounds. Liquid extracts of sisal waste (stumps) have the potential to be used as raw materials for making plastics biodegradable (poly lactic acid) [10].

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Cellulose is one of the most widely distributed and abundant biopolymers on earth, as a major source of renewable materials obtained from plant fiber [11]. Hemicellulose components and lignin-coated nanofibers aggregate into larger bundles, with cellulose becoming an important component of cell walls [12]. In releasing nanocellulose fibers from natural cellulose components such as lignin and hemicellulose through several chemical (acid, base), mechanical (milling, high pressure homogenization, ultra-sonication, high intensity) or enzymatic treatments on cellulose fibrillation from micrometres to nanometers [13].

Various nanocellulose extraction methods have been developed to date, such as mechanical, chemical, and biological methods [14]. Research using chemical methods produces nanocellulose that is quite promising. As in Zhou's research (2012), using the hydrolysis method with strong acids, namely sulfuric acid (H_2SO_4), 64%. At a reaction temperature of 45°C with stirring at 500 rpm for 120 minutes, it produces nanocellulose measuring 115±35 nm [15].

Ioelovich's research (2012) used a hydrolysis method with strong acid, namely sulfuric acid (H_2SO_4). From various variations in reaction temperature and the ratio of acid to the amorphous side of cellulose, the resulting nanocellulose is sized at 150-200 x 10-20 nm [16].

Research by Xiong et al. (2012) used the acid hydrolysis method with strong acid, namely sulfuric acid (63% by weight), as much as 300 ml at 44°C with stirring and ultrasonication at 50 Hz for three hours. The resulting nanocellulose was 10-65 nm in size [17].

Chemical methods, especially using strong acids, can remove the amorphous part of a cellulose chain, thereby isolating the crystalline part of the cellulose.[18]. The quality of nanocellulose produced is influenced by the acid concentration and hydrolysis temperature. As shown by the research that has been done, too high acid concentration will cause cellulose to be damaged as a whole [19]. While lower acid concentrations cannot enter the cellulose matrix to start the hydrolysis reaction [20], temperatures that are too low require a longer time and temperatures that are too high are difficult to control [21]. However, from various studies using this method, it has been able to produce cellulose in the desired size and save energy and operational costs. The main focus of this research is the utilization of sisal fiber decortication waste in extracting cellulose, which is still contained in the waste, using the strong acid method to produce nanocellulose with a nanometer size. Taking into account the appropriate acid concentration and hydrolysis temperature.

Research Methods

Tool and Material

The tools used are analytical scales, grinding machines, pipettes, spatulas, glass funnels, plastic wrapping, hotplate magnetic stirrer, oven, sonicator, centrifuge, measuring flask, chemical bottle, measuring cup, filter paper, and 100 mesh sieve.

The material used is sisal powder *(Agave Sisalana)*, NaOH 2% and 5%, H_2SO_4 65%, $H_2THE_2$3%, and Aquadest.

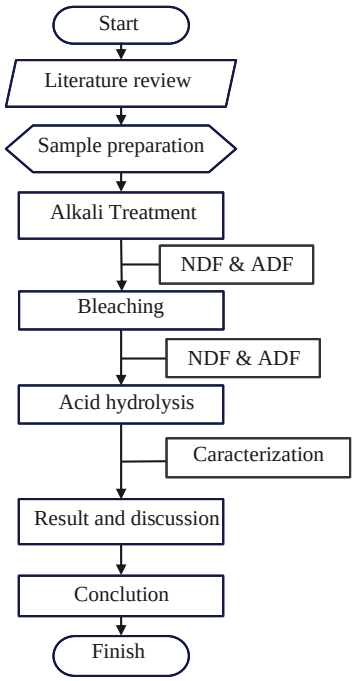


Figure 1. Research Flow Chart

Sample Preparation

The sample used in this study was sisal fiber waste *(Agave Sisalana)*. Sisal is dried under direct sunlight for 3 days. A total of 50 grams of sisal is ground using a grinding machine to a size of 100 mesh.

Alkali Treatment

The sisal fiber is then alkali-treated using NaOH solution with variations of 2% and 5% (1:50) for 2 hours at a temperature of 80 °C at a speed of 200 rpm using a magnetic *stirrer*. After that, the sample was washed with distilled water until the pH was neutral and filtered. Then the residue is dried using an oven for 30 minutes to reduce the water content.

Whitening (*Bleaching)*

Sample results Alkalization Treatment, which has optimal cellulose, hemicellulose and lignin results, is taken and bleached using hydrogen peroxide solution (H2THE23%) at a temperature of 80 °C for 3 hours (1:50). Repeated bleaching once. After that, the sample was washed with distilled water and filtered. Then the residue was dried using an oven at a temperature of 100 °C for 30 minutes.

Acid Hydrolysis

The sample was then subjected to acid hydrolysis using sulfuric acid ($H_2SO_4$65%) at 80 °C for 1 hour (1:20). Then, the sample was diluted with 8 times the initial volume of distilled water to stop the reaction. After that, the residue was dried using an oven at 100^{the}C for 30 minutes.

Sample Characterization

Fiber characterization using NDF to determine the levels of cellulose, hemicellulose and lignin. The results of

the hydrolysis samples were analyzed using FTIR and SEM tests.

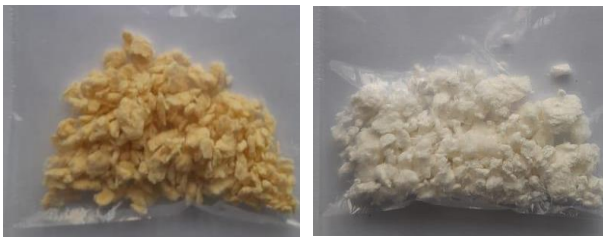
Results and Discussion

Identification of Cellulose Content in Sisal Fiber

NDF testing was conducted to determine the content of sisal fiber decortication waste [22]. Treatment [22]. Alkalization treatment is a process to reduce lignin levels in woody plant fibers, where the lignocellulose structure is opened so that cellulose becomes easier to access [23]. One method of chemical lignin removal uses sodium hydroxide (NaOH). This shows that lignin is the main target for reducing levels. The results of the cellulose content test in sisal fiber decortication waste before treatment were 60.56% after alkalization using 5% NaOH; there was a decrease in cellulose content of 52.21%. The lignin content decreased along with the decrease in cellulose content; there was a significant decrease in the percentage of sisal fiber decortication waste before treatment, with an increase of up to 0.67% after alkalization treatment.

Table 1. Results of Chemical Content Analysis of Sisal Fiber Variations

Fiber variations	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Untreated waste	60.56	24.02	3.44
NaOH 5%	52.21	9.83	0.67



(a) (b)

Figure 2. (a) Sisal waste after alkalization (b) Sisal waste after bleaching

In this study, nanocellulose synthesis was carried out by chemical treatment. The initial process of releasing the lignin layer on the fiber surface. Alkalization treatment using NaOH with attention to stirring and stable temperature so that lignin release can occur optimally, followed by the process Bleachingas many as two repetitions as a method of bleaching or cleaning samples from residual lignin is indicated by a significant color change as shown inPicture 2(a) which was originally yellow turned white (Picture 2(b)) indicates the release of fiber cell walls containing lignin.

Based on the results of Hartono et al (2010), the bleaching treatment of colour changes that occurred using H2THE2, including oxidants that have a fairly strong ability to release oxygen, is environmentally friendly compared to chlorine, and produces stable white cellulose [24]. According to the research results (Lestari and Sari, 2016), which stated that the concentration of H_2THE_2 affects the cellulose produced is the higher the concentration of H_2THE_2thus producing cellulose with a higher degree of whiteness [25]

FTIR Test Spectra Analysis

FTIR spectrum analysis provides an overview of the functional groups of cellulose, hemicellulose and lignin. Cellulose consists of glucose-glucose bonds arranged in a linear chain where C-1 of each glucose is bonded to C-4 of the next glucose.[26]. Figure 3 shows the spectrum results obtained from four different samples, namely untreated samples, alkalization samples, bleaching samples and acid hydrolysis samples.

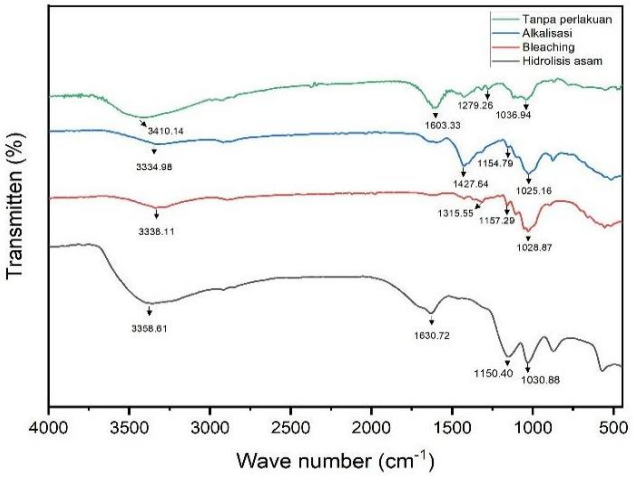


Figure 3. FTIR Spectrum of Variation of Sisal Fiber Waste Treatment

Table 2. Infrared Absorption Region Variation of Sisal Fiber Waste Treatment

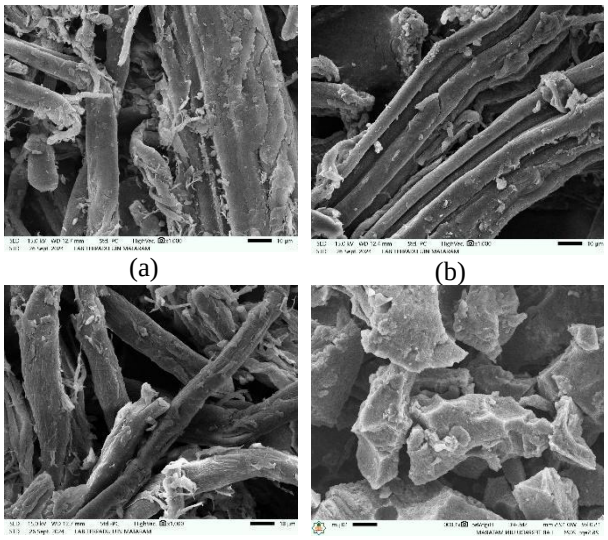
Absorption Area $(cm)^{-1}$)				Bonds and Types of Functional Groups
No Behavior-an	Alkalize-si	Bleach-ing	Acid Hydrolysis	
3410.14	3334.98	3338.11	3358.61	O-H Stretch (s*tretching)*
1603.33	-	-	1630.72	O-H penekukan *(bending)*
-	1427.64	-	-	CH_2 deformation
1315.55	-	-	-	
1279.26	-	-	-	C=C aromatic ring
-	1154.79	1150.90	1150.40	C-O-C Stretch*(stretching)*
1036.94	1025.16	1028.87	1030.88	C-C stretch (*stretching*)

From the FTIR test results, it can be seen from Table 2 that the typical absorption region of cellulose is formed at a wavelength of 3410.14 cm-1, 3334,98 cm-1, 3334,98 cm-1, 3334,98 which indicates the O-H functional group

stretching that occurs at wavelengths ranging from 3500-3000 cm^{-1}[27]. Absorption at wave number 1512 cm-1 is the absorption of C=C aromatic rings typical of lignin, with the peak produced only in the sample without treatment, a wave of 1279.26 cm was formed. This shows that the removal of lignin in the sample has been successful. The C-O-C bond experiences stretching at a wave of 1154.79 cm^{-1}, 1157,29 cm^{-1}, 1150,40 cm^{-1}. In the wave absorption area of 1036.94 cm^{-1}, 1025,16 cm^{-1}, 1028,87 cm^{-1}, and 1030,88 cm-1 show C-C bond stretching [28].

Characterization of Surface Morphology by Scanning Electron Microscopy (SEM)

The fiber is formed by three layers of cell walls, composed of semi-crystalline cellulose microfibrils reinforced by a hemicellulose-lignin matrix with varying compositions. Based on the enlarged SEM image (Picture 4(a)taken from waste from processing sisal fiber without treatment, a layer of lignin is visible on the surface of the fiber. Figure 4(c) from the repeated bleaching fibers, the surface morphology clearly shows very fine nanofibrils with a typical diameter of about several tens of nanometers in accordance with the literature that such microfibrils/nanofibrils have a diameter of about 10-30 nm and consist of 30-100 cellulose molecules in an extended chain conformation and provide mechanical strength to the fibers.



(a) (b) (c) (d)

Figure 4. SEM images of the structure of (a) the untreated sample (b) the alkalization sample (c) the bleaching sample (d) the acid hydrolysis sample

Figure 4 shows a comparison of SEM images (Scanning Electron Microscope) on Gambar (a). The structure is still rough, irregular, and dense. The fibers appear to stick together with lots of dirt/matrix covering the surface. This indicates that lignin, hemicellulose, and other impurities have not been removed. The fiber structure looks more open and fibrous than Picture (b). Some of the matrix begins to disappear, revealing a clearer fiber orientation. This indicates that the alkalization process has removed some of the lignin and hemicellulose. Figure (c)The surface becomes cleaner and smoother. The fibers begin to appear more isolated and individual, indicating that the bleaching process has successfully removed most of the lignin left over from the previous process. In Figure (d), it can be seen that the fiber structure changes drastically, becoming more fragmented and smaller in size (many fine particles are visible). This is an indication that cellulose is degraded, producing a nanocrystalline form or smaller microstructure with a size ranging from 100–500 nm in length, with a diameter of around 5–20 nm.[29] The results of the study showed that nanocellulose with a high % crystallinity was successfully extracted from sisal fiber decoration waste using the acid hydrolysis method. The FTIR spectrum showed a spectrum width of 3358-3410 cm^{-1}which is the vibration of the OH group of cellulose. The removal of lignin levels was successfully carried out, showing that the peak of the spectrum band produced was only 1279.26 cm^{-1}. The average size of nanocellulose particles is around 10-30 nm and consists of 30-100 cellulose molecules [30].

Author's Contribution

Fauzi Widyawati: responsible for conceptualizing the research, developing the methodology, supervising the implementation, writing the initial draft, and reviewing the final manuscript. Syamsul Hidayat: contributed to the technical validation of the acid hydrolysis method. Aditya Wiradana: conducted laboratory experiments, curated data, and assisted in the analysis and interpretation of characterization results. Ayunda Kinasih Setyaningtyas: responsible for the sample preparation stage, data collection, and documentation of experimental procedures. Syamsul Bahtiar: conducted literature review, compiled tables and figures, and edited the manuscript to make it clearer and more consistent. Emsal Yanuar: conducted statistical analysis, interpreted research results, and assisted in the preparation of the discussion and conclusion sections.

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