WASTE CELLULOSE EXTRACTION AND ANALYSIS DECORTICATION SISAL PLANT (*Agave sisalana*) SUMBAWA LABANGKA

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Abstract: Waste utilization of decortication Sisal plants is minimal compared to the amount of waste produced, especially the cellulose content in the waste decortication sishal plant. This research aims to extract waste cellulose from the decortication sishal plants using acid and alkaline solutions with variations in NaOH solutions of 0%, 2%, 4%, 6%, and 8%. The process used is alkalization, bleaching, and acid hydrolysis by method analysis. Next, testing using SEM and FTIR methods is done to determine the morphology of the cellulose content in the resulting waste. Sisal plants are the contributors to the country's foreign exchange in the agricultural sector. Fiber is a type of material in the form of intact elongated tissue. Natural fibers obtained from plants have long been used in various fields of life, such as textiles, ropes, brushes, roofs, handicrafts, and building and construction materials, and materials for making synthetic fibers [2]. In Indonesia, the fiber commonly developed is agave *sisalana* (sisal) and *agave kantala* (kantala).

Sisal fiber, or agave *sisalana* is a natural fiber with relatively high potential in Indonesia. Sisal fiber has chemical components consisting of 78% cellulose, 8% lignin, 10% hemicellulose, and a water content of 10-22% [3]. Sisal has a rosette of sword-shaped leaves about 1.5-2 meters long. Sisal fiber or *Agave sisalana* has been widely applied in various sectors. West Nusa Tenggara Province is currently developing 5,000 hectares of sishal plants around migrant settlements on Sumbawa Island [4], so much sisal and waste will be produced. This can be seen at one of the sisal-producing PTs in Sumbawa, namely PT. Sumbawa Bangkit Sejahtera can produce 44 tons of sisal every day in the production period, where the period for each period is 3 months with a percentage of fiber produced of only 8% and the other 92% is waste, with this percentage in one-period waste production decortication. The production of the sisal plant is 3643.2 tons. In one year, it can produce 7286.4 tons of waste decortication sishal plants in two production periods. However, waste management is still limited decortication. The sisal plants carried out by the PT producing sisal fiber resulted in waste being wasted completely in vain inside waste decortication. It is still possible that the sisal plant has many contents that can be utilized, especially the cellulose content in the waste decortication sishal plant.

According to research, [5] waste decortication Sisal plants contain around 55.7% cellulose, 6.6% lignin and 5.7% hemicellulose. Based on the waste content decortication of the sisal plants presented, it can be seen that the cellulose content has the highest percentage, 55.7%. The potential for this relatively large amount of cellulose will be wasted if no further processing is carried out, which is cellulose in the waste decortication Sisal plants can optimize their utilization and application on certain materials, such as in making surgical threads, biofilms, and others, but there is still little research related to waste extraction. Decortication Sisal and sisal plants prompted researchers to research to test the cellulose content using an alkaline process treatment, bleaching, method Chesson and acid hydrolysis of waste decortication Labangka sisal plant.
Generally, the method often used in the cellulose extraction process is the alkaline method treatment and acid hydrolysis using strong acid and robust base solutions, as for several studies that became the basis for researchers to extract cellulose from sisal waste, namely: [6] Using a refining and high-pressure homogenizing process in the cellulose extraction process in bamboo using strong acid as a destroyer of the amorphous layer of cellulose, the final result obtained from this process is nanocellulose, which is applied to make bio-composites.

The mechanical properties of coconut stem particles depend on the NaOH treatment concentration. With the highest test results in tensile, bending, and impact tests, 4% NaOH was the optimal alkali treatment ratio for coconut stem particles [7].

Researchers the cellulose content in oil palm bunches using the alkalinization method using solid acids and bases. In this research, three processes were carried out in cellulose extraction: alkalinization, bleaching, and acid hydrolysis. The final result is cellulose, with the most cellulose found in oil palm bunches, which undergoes acid hydrolysis [8].

Identified yellow meranti powder waste using Chesson's method to determine the amount of meranti powder using the strong acid H_2SO_4, the results obtained are hemicellulose and cellulose [9].

An analysis was carried out of the effect of NaOH concentration on composite sisal properties. The initial process uses alkali treatment, with the most results obtained in NaOH with a concentration of 5% [10].

The innovation carried out by researchers varied the concentration of NaOH with concentrations of 0%, 2%, 4%, 6%, and 8%.

There is expected to be a lot of cellulose content in the waste decortication. Sisal plants can optimize the use of waste decortication sisal plants. Especially in the application of waste cellulose content decortication micro or nano-sized sisal plants that can be used as surgical threads and become an alternative material for making medical materials that can be degraded in the human body.

**RESEARCH METHODS**

**Material**

The materials used in this research were sisal fiber waste agave *sisalana* from Labangka, West Sumbawa, aquades, H_2O_2 3%, H_2SO_4 98%, and NaOH and waste powder of decortication of sisal plants (*agave sisalana)*.

**Tool**

The tools used in this research are measuring cups and scales, analytical tools, mortar and pestle, pipette, metal spatula, glass funnel, oven, thermocouple, centrifuge, plastic wrapping, magnetic stirrer, hot plate, measuring flask, filter paper, 200 mesh sieve, chemical bottle, mask, and gloves. The tools used to analyze the data are X-ray diffraction (XRD), Scanning Electron Microscope (SEM), and Fourier Transform Infrared (FTIR)

**Preparation of Decortication Waste from Sisal Plants (Agave Sisalana)**

Waste from plant fiber, *Agave sisalana*, was taken from Labangka, Sumbawa Regency, West Nusa Tenggara province. The waste is dried for three days using sunlight and then crushed using mortar and pestle. The ground sample was filtered using a 200 mesh sieve and weighed 1 gram.

**Alkali Treatment**

A sample of the decortication waste from the sisal plant was weighed at one gram. Then it alkalized treatment using four different variations of NaOH concentration, namely 2%, 4%, 6%, and 8% with each variation of one sample for 2 hours 100 ml temperature 80°C at a rotation speed of 200 rpm with the lid closed. The resulting solution was filtered and then placed in the oven for 30 minutes at a temperature of 100°C to reduce the sample's water content.

**Test Chesson**

Decortication waste from sisal plants which has been given through an alkaline process with concentrations of 2%, 4%, 6% 8% NaOH as well as one sample without an alkaline process is then *leaching* using H_2O as a liquid added to waste agave *sisalana* of 150 ml at a temperature of 100°C for 2 hours, the sediment results leaching filtered using filter paper and waited until dry at room temperature, then dry the results *leaching* again using solution H_2SO_4 150 ml with a concentration of 0.5 M for 2 hours at a temperature of 100°C, sediment yield leaching filtered using filter paper and waited until dry at room temperature, then dry the results leaching again using solution H_2SO_4 10 ml with a concentration of 72% for 120 minutes at room temperature, after carrying out leaching with H_2SO_4 72% At room temperature the concentration of the solution was reduced to 4% by adding H_2O and in *leaching* Return for 120 minutes at temperature 100°C each level leaching on method chesson carried out in a closed state, the sediment results leaching filtered using filter paper and washed until the pH is neutral then waited until dry oven with a temperature of 100°C for 30 minutes.

Calculations using formulas for testing cellulose, lignin, hemicellulose, Ash, and water levels.

**Hot Water (%)**

\[ \text{Hot Water (\%) } = \frac{c-a}{a} \times 100 \]  
Equation (1)

**Hemicellulose (%)**

\[ \text{Hemicellulose (\%) } = \frac{b-c}{a} \times 100 \]  
Equation (2)

**Cellulose (%)**

\[ \text{Cellulose (\%) } = \frac{c-a}{a} \times 100 \]  
Equation (3)

**Lignin (%)**

\[ \text{Lignin (\%) } = \frac{d-e}{a} \times 100 \]  
Equation (4)

**Abu (%)**

\[ \text{Abu (\%) } = \frac{b}{1027}\text{a} \times 100 \]  
Equation (5)

Information

\[ a = \text{Initial mass of the sisal plant decortication waste sample (agave sisalana)} \]

\[ b = \text{Mass of precipitate produced at leaching using H_2O} \]
c = Mass of precipitate produced at leaching 0.5 M H₂SO₄
d = Mass of precipitate produced at leaching 72% H₂SO₄ then diluted to 4%
e = Ash from sample residue

Bleaching
Samples that have gone through the test stage chesson with the most significant percentage of cellulose, then weighed 1 gram for the scraping process (bleaching) using 3% hydrogen peroxide (H₂O₂ 3%) 100 ml by doing teaching for 180 minutes and temperature 80°C, then the residue obtained is then placed in the oven for 30 minutes at a temperature of 100°C to reduce the water content of the sample.

Acid Hydrolysis
After the sample is dry, an acid hydrolysis process is carried out. This process uses 100 ml H₂SO₄ 10% for 45 minutes at 60°C in a closed state. Then, the solution obtained is processed by centrifuge to reduce the sample's water content and separate the liquid and solid phases in the sample. In the process, the centrifuge is placed in a microtube, and ensuring the same amount of sample is filled in each tube, then inserted into the tool centrifuge, then rotated at 13,000 rpm for 15 minutes, thus producing a sample in the form of a residue.

Morphological and Functional Group Analysis
Yield residue centrifuge sisal plant decortication waste (agave sisalana), which has been dried, is then characterized using scanning Electron Microscope (SEM) and Fourier Transform Infrared (FTIR).

RESULTS AND DISCUSSION
Sample Preparation
The waste sisal powder used in this research came from the tertiary results of PT Sumbawa Bangkit Sejahtera Labangka's production of sisal fiber, with a rosette of sword-shaped leaves about 1.5-2 meters long. Before leaching using NaOH, samples of the sisal waste are prepared by drying for 3 days using solar heat so that the residual waste is not damp and is not damaged by microbes [11]. After drying, the waste is crushed to a size of 200 mesh. According to [12], increasing the mesh size will reduce the size of the resulting particles. The smaller the particle size, the greater the absorption surface area and absorption capacity. With greater absorption capacity, it is hoped that it will be able to make the sisal waste more reactive so that the hemicellulose and lignin layers can be degraded optimally.

Alkali Treatment
Process alkali treatment aims to remove layers of lignin and hemicellulose. The alkalinization process in this research uses a NaOH alkali tator with varying concentrations of 0, 2, 4, 6, and 8%. Sisal waste that has been alkalinized is then identified using the method Chesson. Based on analysis [14], the highest cellulose content was found in sisal waste treated with 2% NaOH, namely 54% cellulose. The increase in cellulose content was accompanied by a decrease in color density in the sisal waste samples.

In Figure 2, the difference can be seen between the sisal waste that has not been alkalinized treatment and that has been alkalinized treatment. It can be seen in terms of color, and the sisal waste's density decreased after alkali treatment. This shows that the cellulose content is more in the sisal waste than lignin because the dark brownish color indicates the lignin content. This phenomenon also occurred in previous research conducted by [15], which explained that the identification of brown color indicated the lignin content in the sample. Cellulose, lignin, and hemicellulose content in sisal waste after alkaline processing treatment can be seen from the test results in the Table 1.

In Figure 2, the cellulose content obtained decreased significantly, except for the 2% NaOH concentration, which experienced an increase compared to the 0% NaOH concentration, namely an
increase of 23% from 31% to 54% cellulose. This happens because when it is alkaline treatment, NaOH with OH- ions breaks the bonds in the basic structure of lignin, while Na+ binds to lignin to form sodium phenolate. The phenolic salt in this reaction is easily soluble, so in this process, the lignin and hemicellulose will dissolve and pass through the filter paper in a certain amount [8]. Alkaline process reaction treatment can be seen in Figure 2.10. In addition to removing the non-cellulose layer from the surface of the sisal waste, the alkali process treatment is done by reducing the fiber diameter so that the structural relationship between lignin and cellulose is separated [16] different from the 2% NaOH concentration treatment for concentration 4%, 6%, 8% constant decrease. According to [17], who has done pretreatment alkali 1.5% NaOH, cellulose will not be degraded when the NaOH used does not have concentration high temperatures and high temperatures so that the NaOH treatment carried out at 4-8% is considered high enough to degrade the cellulose content in sisal waste. The open structure of cellulose and the free diffusion of cellulose molecules in solution also causes a decrease in cellulose content. So, the dispersed structure of cellulose causes the cellulose to dissolve with the solvent during the filtration process. This triggers a decrease in cellulose content [18].

Moreover, according to [11], lignin increases with increasing concentration in the alkaline process treatment due to the use of solid acid in the test chesson. In acidic conditions, lignin tends to condense so that during deposition, more lignin is isolated [20]. At high acid concentrations, lignin decomposes into monomers, and these monomers react with the lignin remaining in the sisal waste to produce new lignin [17]. This phenomenon causes the lignin content to increase. To analyze the hemicellulose content of the test chesson Quite varied results were obtained. It can be seen that the 0% concentration is 20% of the initial Mass, the 2% and 4% concentrations have a hemicellulose content of 24% of the initial Mass, the 2% and 4% concentrations have a hemicellulose content of 17% of the initial Mass and the 8% concentration has the highest hemicellulose content, namely 40% of initial Mass. According to [17], a higher concentration of NaOH results in a more excellent hemicellulose content, aligning with the treatment carried out.

Bleaching

Process Bleaching is also called a bleaching process that removes residual lignin and hemicellulose after the alkaline process treatment. Bleaching was carried out using H solution:O2; 3% by carrying out the process of bleaching repeatedly, one repetition bleaching. Process repetition bleaching is carried out to increase the purity of the cellulose produced [11]. Figure 4 shows that treatment bleaching affects the color change in the sisal waste. It can be seen that the sisal waste that has been bleached and processed is pale yellow, and the color is lighter than that of the alkaliized sisal waste. This indicates that lignin and hemicellulose

<table>
<thead>
<tr>
<th>No</th>
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<th>Hot Water</th>
<th>Hemicellulose</th>
<th>Selulose</th>
<th>Lignine</th>
<th>Ash</th>
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<tbody>
<tr>
<td>1</td>
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<td>4</td>
<td>2</td>
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</tr>
</tbody>
</table>

Table 1. Test results chesson alkaline treatment and not

Figure 3. Graph of the effect of NaOH concentration on cellulose content
in the sisal waste are decreasing with the lighter color change [21].

Figure 4. (a) Sisal waste treated with 2% NaOH (b) Sisal waste after treatment bleaching.

The decrease in lignin and hemicellulose levels in sisal waste occurs because. The OOH- ion formed in the H2O2 reaction reacts with one group to form a double bond O so that the double bond in benzene is lost and the double bond with O. The missing double bond results in the benzene group becoming unstable. To stabilize the benzene group, the double bond forms a stabilizing double bond, and the O group tends to form a double bond to stabilize the internal benzene group and break bonds with other groups. This reaction removes the C=C double bond. Then, the OH radical group released by OOH- ions reacts and attaches to cellulose so that the concentration of O-H bonds in cellulose increases [8].

Acid Hydrolysis

Acid hydrolysis functions to separate the bonds between cellulose, lignin, and hemicellulose. Acid hydrolysis in this study uses H2SO4 10% for 45 minutes.

Figure 5 (a) Sisal after treatment bleaching (b) sisal waste after acid hydrolysis treatment

Figure 5 compares the sisal waste processed bleaching and acid hydrolysis. It can be seen that the results of samples that have been acid hydrolyzed are brownish yellow, such as sisal waste that has been treated bleaching. However, the acid hydrolysis treatment produces a lighter color than the bleached sisal waste. This indicates that the non-cellulose content in the sample is decreasing [21]. The reduction in non-cellulose content can occur because H3O+ will be produced upon dilution in this process, and the formation of O-H bonds occurs when H ions react with one of the cellulose rings and produces H2O. Then, H2O reacts with other cellulose rings to form O-H bonds and produces H+ ions, increasing the number of O-H bonds and cellulose content [8].

Functional Group Analysis Using FTIR

Analysis of functional groups in cellulose extraction from decortication waste from sisal plants using test equipment. Fourier Transform Infrared (FTIR). The type of FTIR test equipment used is IRPrestige-21 SHIMADZU. The samples tested were samples from each extraction stage, namely samples without treatment, alkali treatment with 2% NaOH, bleaching, and acid hydrolysis. Functional group analysis was carried out to identify the cellulose, lignin, and hemicellulose content at each stage of the extraction process. Identification is carried out based on the wave peaks produced. After adjusting the peaks obtained in a specific wave range, the decortication waste content of the sisal plant will be obtained in each extraction process.

Figure 6. Comparison graph of FTIR results for each test sample

Based on Figure 6 Presented are the results of FTIR testing on four different samples, namely decortication waste from sisal plants without treatment, decortication waste from sisal plants that have gone through an alkaline process with a NaOH concentration of 2%, decortication waste from sisal plants that have gone through an alkaline process with a concentration of 2% NaOH and the process bleaching, and decortication waste from sisal plants which has gone through an alkaline process with a NaOH concentration of 2%, process bleaching and the acid hydrolysis process using H2SO4 10%. The FTIR test results show strong and broad absorption in the 3000-3600 absorption area which indicates the O-H functional group [11] with a peak produced at a wave number of 3410.15 cm⁻¹, 3425.58 cm⁻¹, 3425.58 cm⁻¹, and 3410.15 cm⁻¹. Strong and wider absorption from untreated samples to acid hydrolysis indicates the presence of O-H stretching (stretching) in the hydroxyl group of the cellulose structure.
In each treated and untreated sample of the four samples, only the content in the decortication waste of sisal plants, and following the absorption of the C=C group of lignin. The and alkali treatment.

Table 2. Infrared absorption area of decortication waste from sisal plants without treatment, alkalization, bleaching, and hydrolysis

<table>
<thead>
<tr>
<th>No treatment</th>
<th>Alkali 2%</th>
<th>Bleaching</th>
<th>Acid Hydrolysis</th>
</tr>
</thead>
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<tr>
<td>3410.15</td>
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<td>2854.65</td>
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</table>

Figure 6 also explains the hemicellulose content contained in each sample. The bonds that indicate the presence of hemicellulose are C-H bonds in the range 2800-3000 cm\(^{-1}\) [22]. In each treated and untreated sample, a peak indicates the presence of hemicellulose content, which can be seen in the area 2846-2986 cm\(^{-1}\). This shows that there is still hemicellulose content in the decortication waste from sisal plants that have been treated. What indicates the lignin content in the graph is the C=C double bond; this bond is in the range of 1200-1300 cm\(^{-1}\) [23]. Of the four samples, only the untreated sample had lignin content, namely at the peak of 1280.73 cm\(^{-1}\), for samples that have been alkaliized at bleaching and acid hydrolysis do not have C=C lignin double bonds because it does not have a peak in the range 1200-1300 cm\(^{-1}\) which indicates the removal of lignin content.

Table 2 shows the infrared absorption area of decortication waste from sisal plants without treatment, decortication waste from sisal plants that have gone through an alkaline process with a concentration of 2% NaOH, waste from decortication from sisal plants that have gone through an alkaline process with a concentration of 2% NaOH and the process bleaching, and decortication waste from sisal plants which has gone through an alkaline process with a NaOH concentration of 2%, process bleaching and acid hydrolysis process. Alkaline process treatment, which has been carried out, increases the concentration of the O-H functional group, indicated by a peak in the absorption area between 3200-3600 cm\(^{-1}\) compared with untreated sisal plant decortication waste. The absorption area around 1590 cm\(^{-1}\) shows bending cellulose [24] and undergoes increased O-H bonding and alkali treatment. This also functions to eliminate the absorption of the C=C group of lignin. The following treatment is bleaching, which removes lignin content in the decortication waste of sisal plants, and this can be seen from the C=C bonds that disappear in the FTIR test results. The third treatment is acid hydrolysis. In this treatment, no C=C bonds were found, which shows that the acid hydrolysis process can remove quite a lot of lignin in the decortication waste from the sisal plant.

Morphological Analysis Using SEM

Morphological analysis of cellulose extraction from decortication waste from sisal plants using test equipment. Scanning Electron Microscopy (SEM). The samples tested are samples that have passed the alkaline stage treatment with 2% NaOH, bleaching, and acid hydrolysis; morphological analysis is carried out to identify the sample surface topography and composition contained in the sample. Identification is carried out by analyzing the topographic image of the sample surface obtained from the test results. The results obtained are adjusted to the characteristics of the position of cellulose, lignin and hemicellulose in plants.

![Figure 7](image-url) Figure 7. The results of observations of cellulose waste that has gone through an alkaline process, bleaching, and acid hydrolysis.
Figure 7 shows the morphology of the decortication waste from sisal plants treated with alkali treatment, bleaching, and acid hydrolysis. Figure 7 shows that the structure of the decortication waste from the sisal plant is already loose. The loose structure can be seen from the layers of lignin and hemicellulose peeling off and separating from the cellulose. The peeling of lignin and hemicellulose in the cell walls of the decortication waste from the sisal plant resulted in the structure of the decortication waste from the sisal plant starting to weaken and its surface degraded. This shows that the treatment that has been carried out causes the separation of bonds between cellulose, lignin, and hemicellulose [25]. So, separating these bonds produces cellulose with an average diameter of 28.64762 µm and 33.6 9369 µm. The reason the cellulose produced does not reach nano size is that it is suspected that the acid concentration used in the acid hydrolysis process is still too small, which results in the function of separating the bonds of lignin, hemicellulose, and cellulose in acid hydrolysis [8]. is still not optimal so the cellulose obtained is in micro size.

CONCLUSION
Morphological and chemical group testing that has been carried out on the decortication waste powder from the sisal plant can be concluded that the cellulose extraction process begins by drying the waste decortication sisal plants and reducing the size of waste decortication sisal plants up to 200 mesh size, the alkalized using 2% NaOH for 2 hours using a temperature of 80°C then bleaching with H2O2 3% use a temperature of 80°C for 3 hours and acid hydrolyzed with H2SO4 10% uses a temperature of 60°C and centrifuged at a speed of 13,000 rpm for 15 minutes. The influence of the alkaline process, bleaching, and acid hydrolysis in the research is to increase the amount of cellulose by removing the non-cellular layer in the waste decortication. In terms of color, the treatment carried out in Sisal plants produces waste decortication. Sisal plants are brighter in brownish-yellow color compared to waste decortication sisal plants without treatment. The cellulose obtained was microcellulose with sizes of 28.64762 µm and 33.6 9369 µm. The functional group test results show alkaline treatment, bleaching, and the acid hydrolysis carried out shows an increasingly stronger and broader O-H peak the adsorption, and eliminates lignin C=O bonds in waste decortication sisal plants, strong and wider absorption from samples without treatment to acid hydrolysis indicates the presence of O-H stretching (stretching) in the hydroxyl group of the cellulose structure. The disappearance of C=O bonds, which indicates a reduction in the amount of lignin in the waste decortication sisal plants, is directly proportional to the results of morphological tests, which show the structure of the waste fiber decortication Sisal plants are already stretched with layers of lignin and hemicellulose peeling away from the cellulose.

REFERENCES