CHEMICAL CONTENT AND ANATOMICAL CHARACTERISTICS OF SAGO (*Metroxylon sagu* Rottb.) FROND FROM SOUTH KALIMANTAN, INDONESIA

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CHEMICAL CONTENT AND ANATOMICAL CHARACTERISTICS OF SAGO (*Metroxylon sagu* Rottb.) FROND FROM SOUTH KALIMANTAN, INDONESIA. This research aims to evaluate the suitability of sago palm waste as a fiber raw material in terms of its chemical content and anatomical characteristics. The chemical content analysis of its extractive components, lignin, holocellulose, α-cellulose, and hemicellulose, was carried out using sago frond powder with a size of 40–60 mesh. Subsequently, functional group analysis was performed using Fourier Transform Infra-Red (FTIR), while anatomical characterization was carried out by calculating the fiber length and diameter, lumen diameter, fiber derivative values, and wall thickness using a microscope connected to a digital camera. Scanning Electron Microscope (SEM) pictures were taken in different magnifications. The results showed that sago frond contains 31.6% α-cellulose and 38% lignin. The α-cellulose content was within the standard range for non-timber forest products, but the lignin content had a relatively high value. Based on the derived value, namely class II, sago frond can be used as pulp and paper raw materials.

Keywords: Sago palm, *Metroxylon sagu* Rottb., anatomical characteristic, chemical content, FTIR

*KANDUNGAN KIMIA DAN KARAKTERISTIK ANATOMI PELEPAHSAGU (*Metroxylon sagu *Rottb.) DARI KALIMANTAN SELATAN. Tujuan dari penelitian ini adalah untuk menganalisis kesesuaian limbah tanaman sagu (pelepah sagu) sebagai bahan baku pulp dan kertas berdasarkan kandungan kimia dan karakteristik anatominya. Serbuk pelepah bukuran 40–60 mesh digunakan dalam proses analisis kimia kayu. Kandungan kimia berupa ekstraktif, holoselulosa, α-Selulosa, dan hemiselulosa dan kadar lignin dianalisis dan selanjutnya dianalisis menggunakan Fourier Transform Infra-Red (FTIR). Penghitungan panjang dan diameter serat, diameter lumen, dan tebal dinding sel diukur untuk karakteristik anatomi selanjutnya perhitungan nilai turunan serat dihitung berdasar data yang didapat. Pengambilan gambar dengan beberapa perbesaran yang berbeda menggunakan Scanning Electron Microscop (SEM) dilakukan. Dari penelitian diperoleh data bahwa kadar α-selulosa dan kandungan lignin secara berurutan sebesar 31,585% dan 37,996%. Kadar ligninnya menunjukkan nilai yang relatif tinggi namun kandungan α-selulosa pelepah sagu menunjukkan nilai standar pada produk hasil hutan bukan kayu. Dari nilai turunan serat pelepah sagu tergolong kelas II sehingga dinilai memiliki kesesuaian untuk bahan baku pulp dan kertas.*

Kata kunci: Sagu, Metroxylon sagu *Rottb., karakteristik anatomi, kandungan kimia, FTIR*

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I. INTRODUCTION

Sago plants (*Metroxylon sagu* Rottb.) are classified as abundant non-timber forest products in Kalimantan, where they grow in swamps and along river banks. In 2022, the plantation area in South Kalimantan was 7,857 ha, with a total production of 4,511 tons ha^{-1} (Saputra, Kissinger, & Itta, 2022). The Sago plant has tall stems with a wide diameter. During its processing to obtain flour, some wastes are often produced from the parts which have not been optimally used, namely the frond and bark. The remaining part is a source of cellulose as a raw material for the pulp and paper industry.

Fiber sources as raw materials for pulp can be divided into two groups, namely wood and non-wood types. In Indonesia, the pulp industry often obtains its raw materials from broadleaf wood. The fibers produced by these species are relatively short, with a range of 0.76-2.9 mm (El-Sayed, El-Sakhawy, & El-Sakhawy, 2020). Short fibers often produce a homogeneous and perfectly mixed pulp; hence, they can be used to manufacture paper with a smooth surface, high opacity, improved printing quality, and high flexibility. However, these papers have low strength, specifically in their tear and tensile indexes (Fiserova, Gigac, & Balbercak, 2009; Yahya et al., 2019). Adding long fibers to the pulp mixture is necessary to increase the paper's strength. Long fibers can be obtained from needle-leaf wood and non-timber forest products, such as frond, bagasse, bamboo, jute, straw, and agricultural waste.

Sago frond and bark are abundant sources of cellulose in South Kalimantan. Several studies were also carried out on the wastes, such as the application of the frond as an alternative adsorbent material and charcoal (Wahi, Chuah, Ngaini, Nourouzi, & Choong, 2014; Johan, Ahmed, Omar, & Hasbullah, 2021), but none explored its usage as an alternative mixture for raw materials in the particle board and pulp and paper industry. The major component of sago frond is crude fiber and carbohydrates, with a value of 17.90%-34.44% and 51.44%-72.87%, respectively (Marvie & Sunarti, 2021). Therefore, an analysis of its chemical content and anatomical characteristics as a source for pulp and paper raw materials and particle board was carried out in this study. The use of the waste can increase their value of sago frond.

II. MATERIAL AND METHOD

A. Materials

The material used for this research was frond of sago (*M. sagu* Rottb.), acetic acid (CH^3COOH), sulfuric acid (H_2SO_4), benzene (C_6H_6), nitrite acid (HNO_3), acetone (CH_3COH_3), ethanol (C_2H_5OH), 1% and 17,5% of natrium hydroxide, distilled water, xylol, and safranin. The equipment used in this study was an electric microscope, desiccator, hot plate stirrer, magnetic stirrer, electronic balancing, water bath, hammer mill, 40 and 60 mesh sieve, oven, beaker glass, erlenmeyer, object and cover glass.

B. Procedure

The sago frond waste was collected from Sungai Tabuk village, Banjar Regency, South Kalimantan, Indonesia. About 2 m of sago frond from the base were collected. It was produced in the form of chips with a size of 3 cm x 3 cm x 2 mm and then powdered. The chemical content analysis and Fourier Transform Infra-Red (FTIR) (Shimadzu FTIR Prestige-21) were carried out using powder with a size of 40 - 60 mesh. The sample was placed in an FTIR spectrometer, and then the FTIR instrument sends infrared radiation of about 10.000 to 100 cm-1 through a sample, with some radiation absorbed and some passed through. The absorbed radiation is converted into vibrational energy. The resulting signal presented as a spectrum representing a molecular fingerprint of the sample at the detector. The chemical structure can be identified due to each chemical structure will produce a unique spectral fingerprint. Furthermore, the sample used for the measurement of fiber dimensions was in the form of a stick, which was macerated using

the Schultze method. The softened sample was stained with safranin and washed in alcohol with a graded concentration, followed by immersion in xylol.

Moisture Content

The sample was weighed and placed in a weighing bottle with a known weight. It was then dried at 103 ± 2°C for 12 hours until it was constant in the oven. Moisture content (MC) was then calculated using the formula (1):

$$MC = \frac{Innitial\ mass - Oven\ dry\ mass}{Oven\ dry\ mass} \times 100\% \quad (1)$$

Determination of Chemical Components

a. Extractive Content in Ethanol – Benzene

To measure the extractive content, a 2.5 g sample was extracted with a 120 mL mixture of 95% ethanol and benzene (1: 2, v : v) for 6 hours using a Soxhlet extractor. The extractive content dissolved in benzene ethanol was calculated using standard methods (JWRS, 2000).

b. Lignin Content

The lignin content was obtained from the extractive-free samples, which were placed in a beaker. Subsequently, 30 mL of 98% H_2SO_4 was added and the mixture was kept at room temperature for one day. The solution was then boiled at 100°C for 60 minutes while the filtrate was washed until the smell of sulfuric acid disappeared. The constant weight can be found by dried the samples in an oven at 103 ± 2°C. The residue obtained from the extraction was the lignin content.

c. Holocellulose Content

One gram of extractive-free sample was placed into a 100 ml Erlenmeyer flask after weighed. Its MC was then measured and used to calculate the holocellulose content. Furthermore, 1.5 ml of 25% $NaClO_2$ (sodium chlorite), 0.125 ml of 100% glacial acetic acid and 40 ml of distilled water were added to the sample. The mixture was stirred, and the Erlenmeyer flask was tightly closed, followed by heating in a water bath at 80°C for 60 minutes. It was then cooled in an ice bath and filtered using a weighed 1G3 filter glass. A total of 10 ml sample was added, followed by 25 ml of acetone. It was then dried for 1 day in an oven at 103 ± 2°C, after which it was cooled in a desiccator for 30 minutes and weighed.

d. α-Cellulose Content

The test of α-cellulose content in sago frond and bark was carried out in different steps. An empty 1G3 funnel filter was dried at 103 ± 2°C in an oven, placed 30 minutes in a desiccator, and the oven-dry weight was measured. Holocellulose sample was weighed (0.5 g) and placed in a ± 20 mL wide-mouth vial. Subsequently, 17% NaOH (6.25 ml) was added, followed by stirring using a for 15 minutes using magnetic stirrer. The mixture was then left for 30 minutes, and 8.25 ml of 17% NaOH was added. It was stirred using a magnetic stirrer for 5 minutes and left for 60 minutes. The final mixture was then filtered using a IG3 filter glass, followed by rinsing with 8.3% NaOH (100 ml) and 25 ml of distilled water. The hose attached to the vacuum bottle was removed, and the sample in IG3 was placed in 10 ml of 10% acetic acid for 3 minutes. Subsequently, the hose was reconnected and left until the entire solution was sucked out. The sample was then washed with distilled water until it became neutral, as indicated by the blue litmus paper attached. It was dried in an oven at 103 ± 2°C for 24 hours, removed, cooled in a desiccator for 30 minutes, and weighed

e. Hemicellulose Content

The hemicellulose content was calculated by subtracting the holocellulose content from α-cellulose.

Measurement of Fiber Dimension and Derived Fiber

Small sticks of sago frond were collected and macerated with Schulz's solution as preparation to determine sago fiber anatomical properties. Fiber dimensions of fifty fibers

were measured under an electronic microscope and used to determine fibers properties. Runkel Ratio, Slenderness Ratio, Muhlsteph's Ratio, Coefficient of Rigidity, and Flexibility Ratio were calculated by the following equation (Istikowati el al., 2016a):

$$Runkel\ Ratio = \frac{Fiber\ wall\ thickness \times 2}{Fiber\ lumen\ diameter} \quad (2)$$

$$lenderness\ Ratio = \frac{Fiber\ length}{Fiber\ diameter} \quad (3)$$

$$Muhlsteph's\ Ratio = \frac{(fiber\ diameter)^2 - (fiber\ lumen\ diameter)^2}{(fiber\ diameter)^2} \quad (4)$$

$$Coefficient\ of\ Rigidity = \frac{Fiber\ wall\ tickness}{Fiber\ diameter} \quad (5)$$

$$lexilility\ coefficient = \frac{Fiber\ Lumen\ diameter}{Fiber\ diameter} \quad (6)$$

The Scanning Electron Microscope figures frond were then taken in different magnifications.

III. RESULT AND DISCUSSION

The results of chemical content analysis the sago frond showed the moisture (%), ethanol-benzene extractive (%), lignin (%), acid-soluble lignin (%), holocellulose (%), and ∝-cellulose (%) content as well as the FTIR.

A. Chemical content analysis

The moisture content (MC) of the sago frond was 75.434%. The value obtained can vary in different areas with the same plant species. Moisture content can also be affected by the dryness degree of the powdered sample before it is dried using the oven. Water is needed by plants to transport nutrients and minerals. Determination of MC in the pulp and paper industry is used to calculate the consume of cooking chemicals in the pulping process.

The ethanol-benzene extractive content of the sago frond was 5.56% (Table 1), which was lower compared to that of Purun Tikus (*Eleocharis dulcis*) extract, namely 9.53%. High value of ethanol-benzene extractive in pulp raw materials is not expected because it causes difficulty in the breakdown of fiber during the cooking process (Sunardi & Istikowati, 2012). The low extractive content of sago frond makes it suitable as a pulp and paper raw material and fiber-based product such as fiber biocomposite. It is because extractive substances can cause pitch, namely spots on the paper produced, and the tools used can easily be dull. High levels can also inhibit the entry of chemicals during the pulp ripening process (Sugesty, Kardiansyah, & Pratiwi, 2015; Istikowati et al., 2016b).

Furthermore, the lignin content of sago frond was 37.99% (Table 1), which was higher than *Pandanus helicopus* and almost the same as reeds lignin, namely 31.67% and 31.29%, respectively. The value obtained was also higher compared to woods usually used as raw material for pulp and paper, namely *Acacia mangium*, *Falcataria moluccana*, and *Eucalyptus*

Table 1 Chemical content of non-wood materials

Parameters	Chemical content (%)				
	Sago (*Metroxylon sagu*) frond	Purun Tikus (*Eleocharis dulcis*)[1]	*Pandan rasau (Pandanus helicopus*[2])	Salacca (*Salacca zalacca*) frond[3]	Nipa (*Nypa fruticans*) frond[4]
Moisture content	75.43	92.68	96.07	-	-
Extractive content	5.56	9.53	4.60	7.8	1.9
Lignin content	37.99	26.40	31.67	23	17.8
Holocellulosa	55.63	-	58.73	57.6	61.6
α-Cellulosa	31.59	-	27.06	29.3	35.1
Hemicellulose	24.05	-	31.67	28.3	26.4

Notes: [1] Sunardi & Istikowati (2012); [2] Herlina et al. (2019); [3] Hakim et al. (2021); [4] Tamunaidu & Saka (2011)

urophylla with content of 31.30%, 23.77%, and 24.31%, respectively (Karlinasari, Nawawi, & Widyani, 2010; Yahya et al., 2019; Nasdy, 2013). Sago frond lignin content can be categorized as high, which is not needed in the industry because it can increase the need for cooking chemicals, thereby making it less economical (Putra, Wardenaar, & Hasni, 2018). Lignin is a component that must be removed in the pulping process, making the wood cells break down easily into single fibers. Its presence in the pulp can inhibit hydrogen bonding, cause an adverse effect on color, and increase the stiffness of the paper sheets (Sugesty et al., 2015). Material with low extractive and lignin content is more suitable for raw material of pulp because the content affects pulp yield as well as the bleaching process, with higher content of extractive and lignin in wood leading to lower pulp yield and paper strength (Istikowati et al., 2016b).

High α-cellulose content is needed in the manufacture of pulp because it increases the yield as well as holocellulose (Yahya et al., 2019). The value of holocellulose and α-cellulose obtained for sago fronds in this study was 55.63% and 31.59%, respectively. Cellulose-based materials with high holocellulose content are good for pulp and paper because they help to increase the yields during the pulping process.

Hemicellulose acts as a binder to the paper-forming fibers; hence, a sufficient amount helps to produce quality papers (Yahya et al., 2019). The pulp yield and physical properties of the sheet produced are often affected by the hemicellulose content. However, very high levels are not suitable for the pulping process because the time and power required for milling and separating the fibers during mechanical treatment also increase (Sugesty et al., 2015).

B. Fourier Transform Infra-Red (FTIR)

FTIR is a spectrum measurement technique based on the material's response to electromagnetic radiation. It is often used for qualitative and quantitative analysis to determine the groups of organic and inorganic compounds in a sample. The measurement can also be used to determine the molecular structure of a compound. The results of the FTIR wave spectrum on the extractive-free sago frond are presented in Figure 1.

Functional group analysis was carried out to determine the holocellulose, α-cellulose, hemicellulose, and lignin content of the sample. Furthermore, the groups in organic compounds can absorb electromagnetic radiation at wavelengths of 2.5-25 m or wave numbers of 400-4000 cm^{-1} (Sutiya, Istikowati, & Rahmadi, 2012). The data of the IR spectrum wave and its interpretation are presented in Tables 2 and Table 3, respectively.

The O-H functional group is the cellulose hydroxyl obtained from the spectrum of the sample, as shown in Table 3. The C=C group extends the aromatic ring (lignin) to the sago

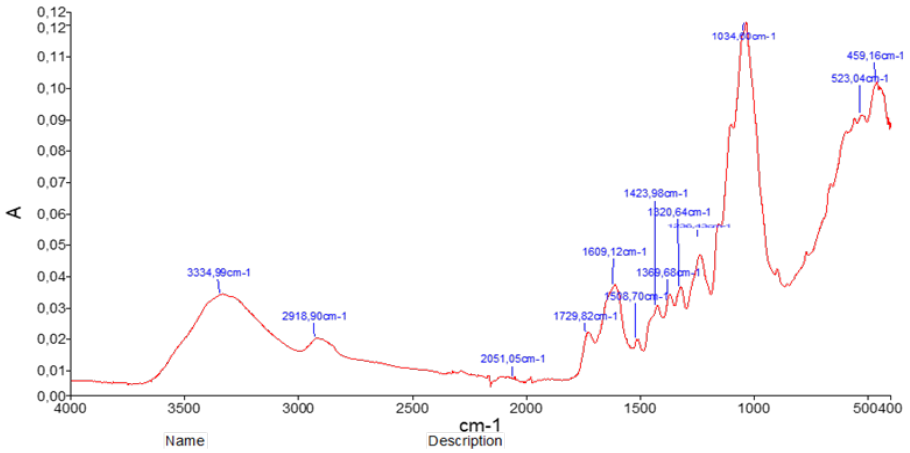


Figure 1. FTIR spectrum of sago frond

frond. The wave number for the C-H bonding of cellulose, hemicellulose, and pectin was 459.16 cm^{-1}.

FTIR presents qualitative data in the form of a spectrum that shows peaks or wave numbers. The wave numbers, which were interpreted into functional groups and chemical bonds, are used to determine the chemical content of a material. The spectrum of the sago frond shows its lignin, hemicellulose, and cellulose content. The extractive wave value does not appear in the FTIR test because it uses an extractive-free sample. The results are in line with the chemical component tests that sago frond contains 37.99% lignin, 55.63% holocellulose, 24.05% hemicellulose, and 31.59% α-cellulose.

C. Anatomical Characteristics

Dimensions (fiber length and fiber diameter, lumen diameter, and cell wall thickness) of the sago frond measured using microscope. The results of the average measurement of fiber dimensions were used to obtain its derivative value (Runkel ratio, muhsteph ratio, slenderness, coefficient of rigidity (stiffness), and ratio of flexibility), and they have a positive correlation. The results of the dimension measurement of the sago bark and frond fiber are presented in Table 4.

Table 2. Wave spectrum of sago frond

No.	Wave number (cm^{-1})	Functional group
1.	3,300	O-H
2.	3,400	N-H
3.	<3,000	C-H sp^3
4.	>3,000	C-H sp^2
5.	3,300	C-H sp
6.	2,850 & 2,750	C-H aldehyde
7.	2,250	C=N
8.	2,100	C=C
9.	1,300-1,000	C-O
10.	800-600	C-Cl
11.	~1,460	CH_2 bending
12.	~1,380	CH_3 bending

Source: Durmas et al., 2016

Table 3. Interpretation of infra-red wave spectrum

Spectrum of sago frond	Note
3,334.99	Shows the O-H functional group of the hydroxyl group (α-cellulose)
2,918.90	Shows C-H from the metal group
2,051.05	C=C Aromatic ring group (lignin)
1,729.82	C=O Acelyt group, carboxylic acid (hemicellulose)
1,609.12	C=C Aromatic ring group (lignin)
1,508.70	C=C Aromatic ring group (lignin)
1,423.98	C-H deformation (lignin, hemicellulose)
1,369.68	C-H vibration (α-cellulose)
1,320.64	C-H vibration (α-cellulose)
2,361.43	-
1,034.60	Shows C-O vibration from β-1,4-glycoside bonding (α-cellulose)
523.04	C-H deformation (lignin, hemicellulose, pectin)
459.16	C-H deformation (lignin, hemicellulose, pectin)

Source: Durmas et al., 2016

Sago frond is a lignocellulosic natural fiber with a long length, and it is longer than P. helicopus and Purun tikus, while it has almost the same length as reed fibers. Long fibers often have stronger bonds, and they are not easily separated. Furthermore, the folding strength of the paper was high, and it was not easy to tear. Excessively long fibers can lead to the production of rough paper. Hence, it is important to reduce the length to increase the smoothness of the product.

The diameter of the sago frond fiber was 12.77 μm, and it was classified as slender. It indicates that the sample is suitable for pulp because it can produce thinner and stronger paper (Sunardi & Istikowati, 2012). The slender fibers are easily interwoven and form sheets of paper with good properties, which are not easily torn.

The lumen diameter of the sago frond was 12.77 μm, and this value is higher than Purun tikus and bamboo Betung but lower compared to reeds. The fibers also have thinner cell walls compared to reeds and bamboo betung. Thin walls are easily flattened, providing a large surface area for bonding between fibers. This condition causes low tear values but high folding, breaking, and tensile strengths (Yahya et al., 2019).

D. Derived Wood Properties

Derived wood properties were obtained by comparing the results of the dimension measurements. Derived wood was used to predict the quality of the pulp to be produced. Moreover, the derived values of sago bark and frond are presented in Table 5. The requirements and value of wood fiber as pulp and paper raw material (Table 6). The average fiber length of sago frond fibers is 2.11 mm, which can be categorized as class II. The fiber length of the sago frond is almost similar to reed and longer than Pandan Rasau and Purun Tikus, as shown in Table 4. The length of fiber produce paper with high strength quality.

Table 4. Fiber dimension of non-wood materials

Type	Fiber dimension			
	Fiber length (mm)	Fiber diameter (μm)	Lumen diameter (μm)	Fiber wall thickness (μm)
Sago frond	2.11	12.77	5.92	3.61
Pandan rasau[1]	1.56	11.10	6.80	2.50
Purun tikus[2]	1.68	5.89	2.68	1.61
Palm oil[3]	1.07	28.15	22.57	2.79

Notes: [1] Herlina et al. (2019); [2] Sunardi & Istikowati (2012); [3] Yahya et al. (2019)

Table 5. Derived wood properties of non-wood fiber

Derived wood	Sago (*Metroxylon sagu*) frond	Pandan rasau (*Pandanus heliocopus*)[1]	Purun Tikus (*Eleocharis dulcis*)[2]
Runkel ratio	1.30	0.72	1.20
Slenderness ratio	164.50	140.54	285.45
Muhsteph ratio (%)	81.24	166.46	38.40
Coefficient of rigidity	0.28	0.2	0.27
Flexibility ratio	0.43	0.61	0.45

Notes: [1] Herlina et al. (2019); [2] Sunardi & Istikowati (2012)

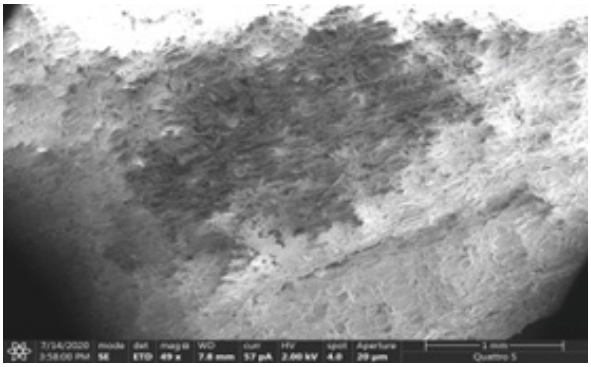
The Runkel ratio of sago frond was 1.30, and it can be categorized as class IV. A small ratio is important during the manufacturing process because it produces paper with strong fiber bonds and flat sheets, while a high value reduces flexibility and produces stiff paper (Istikowati et al., 2016b).

The slenderness ratio of the sago frond was 164.50; hence, it can be categorized as class 1. The value obtained was higher than pandan rasau, purun tikus, and reed fibers. Sago frond has a long fiber, which makes the weaving power and bonding very strong. Furthermore, a high value is related to the smoothness of the paper produced. High weaving power has a good influence on the tensile strength and breakage of paper or filler in biocomposites (Sugesty et al., 2015).

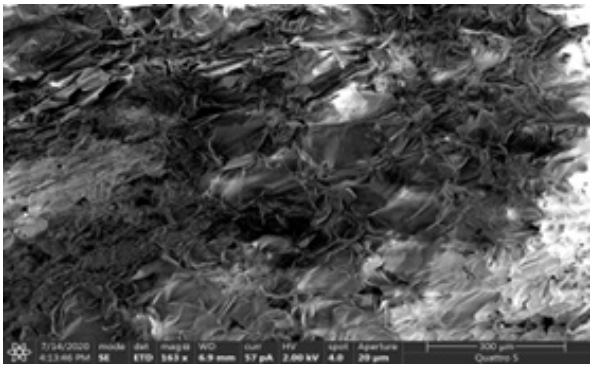
The Muhsteph ratio of sago frond was 81.24%, and it was categorized as class III.

Table 6. Standard of derived wood properties (Silitonga, 1972)

	Class I		Class II		Class III		Class IV	
	Requirement	Score	Requirement	Score	Requirement	Score	Requirement	Score
Fiber length (mm)	2.2	100	1.6-2.2	75	0.9-1.6	50	<0.9	25
Runkel ratio	<0.25	100	0.25-0.5	75	0.5-1.0	50	>1.00	25
Mulhstep ratio (%)	<30	100	30-60	75	60-80	50	>80	25
Slenderness ratio	>90	100	70-90	75	40-70	50	<40	25
Flexibility ratio	>0.80	100	0.6-0.8	75	0.4-0.6	50	<0.40	25
Stiffness ratio	<0.1	100	0.1-0.15	75	0.15-0.2	50	>0.20	25
Total		600		450		300		50
Score requirement	451-600		301-450		151-300		150	



(A)



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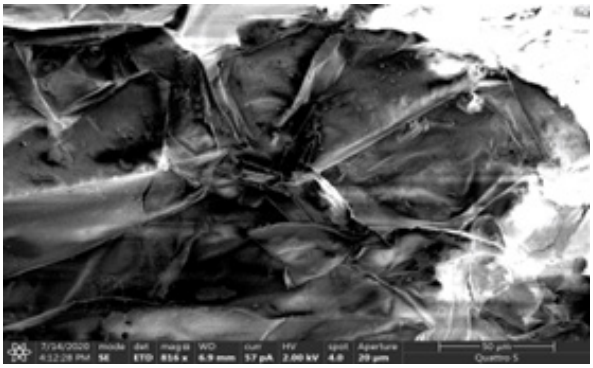

(C)

Remarks: A, B, C = SEM of sago frond with magnification 30, 100, dan 500, respectively

Figure 2. Scanning Electron Microscope (SEM) of sago frond

Paper produced from a high ratio has a rough surface, low squeezing strength, and is not plastic. Based on the value obtained, the fibers are not suitable for writing paper, but they can be used to produce wrapping or art paper. The coefficient of rigidity was 0.28, and it was categorized in class IV. The magnitude of this value is related to the stiffness of the paper produced. The higher the coefficient of rigidity, the higher the stiffness. The paper produced from these fibers is expected to have low tensile strength.

The flexibility ratio obtained was 0.43, and it was categorized in class III. A high ratio can produce papers that are more flexible and less stiff. The overall value of sago frond fiber derivatives was 325, and it was categorized in class II. Based on their properties, they can be used as an alternative raw material for pulp and paper.

The observation under SEM at high magnifications showed the 3D structure of the sago frond in different magnifications. Morphological characterization of the sago frond using SEM aims to observe the surface roughness. Based on the SEM photo obtained, the surface shows many cavities with thin cell walls (Figure 2). SEM images of the sago frond showed the cells of sago frond; however, it was not possible to differentiate between fiber and parenchyma

IV. CONCLUSION

The α-cellulose and lignin content of the sago frond were 31.59% and 37.99%, respectively. Furthermore, the amount of the lignin was relatively high but α-cellulose in the sample showed a standard value in non-timber forest products. The sago palm was classified as class II based on the fiber derivatives. This indicates that it is suitable as pulp and paper raw and product-based fiber material but needs pretreatment for lignin removal.

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