Delignification and Characterization of Fiber from Durian Peel Waste

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Abstract. The limited availability of natural fiber sources makes durian peel waste an alternative source of natural fiber. The characteristic of durian peel waste, which is mechanically strength, has the potential to be developed. During the durian season, the amount of durian consumption by the community increases so that the amount of durian skin waste produced will also increase. It raises environmental problems. Therefore, the research was conducted to study the potential of durian peel fiber as a source of textile fiber. The research carried out several stages, including physical and mechanical initial treatment and a chemical delignification process using an alkaline solution. The crude fiber obtained is then bleached, dried, and analyzed for textile fiber applications. The analysis included fiber content using the Chesson-Datta method, dimensional analysis using Scanning Electron Microscopy, strength, elongation, and fineness (micronaire) analysis. According to analysis, durian skin fiber is rough, brittle and unsuitable for clothing fiber applications. However, it still can apply for other textile applications.

Keywords: Durian Skin, Delignification, Cellulose.

1 Introduction

Fiber is a long, thin, and easily bent material with a ratio of length to width reaching several hundred. Fiber comprises macromolecules such as cellulose, protein, and minerals [1]. It can be classified as natural, semi-synthetic, and synthetic fiber. Natural fiber is obtained from plants and animals, while synthetic fibers are produced physically or chemically. Otherwise, semi-synthetic fibers are a combination of natural and synthetic fibers. Fiber has received much attention from researchers because the need for textiles will continue to increase in the next few years and reach nearly 133.5 million tons in 2030. It is due to increased economic growth and population numbers [2].

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Natural fiber sources, including cotton, silkworms, and others, are limited. Due to the limited availability of natural sources, researchers are trying to find alternative materials that can be used as fiber sources. Many fiber sources have been used, such as pineapple, corn fronds, and others. One potential alternative fiber source is durian skin waste. The durian peel waste has mechanical strength and potential to be developed. Durian fruit (*Durio zibenthinus*) is a fruit that is popular in Southeast Asia, especially in Indonesia. In Indonesia, there are 103 varieties of durian fruit [3]. During the durian season, the amount of durian consumption by the community increases so that the amount of durian skin waste produced will also increase. According to research from the Central Bureau of Statistics in 2020, local durian production in North Sumatra reached 74,675 tons yearly. 40-42% of it is durian skin waste [4]. It will contribute to an increase in organic waste in Indonesia. Therefore, it is necessary to minimize durian skin waste by utilizing it for valuable products.

Although durian peel waste has been used as bioethanol [5], bio composite [6], briquettes [7], and vegetable biopesticides [8], the amount of durian skin waste is still a cause of organic solid waste problems. One alternative solution to the problem is utilizing durian peel waste as a raw textile material. The advantage of fiber from durian skin is that it is easily degraded compared to synthetic fibers because cellulose reaches 33-57% [9,10]. Natural fibers are more comfortable to use and environmentally friendly than synthetic fibers [2], but due to low production rates, limited availability, and high demand, natural fibers are expensive.

For the isolation of durian peel waste fiber, the delignification process is critical. The delignification process is a process to remove the lignin contained in the biomass. Lignin is a phenolic component in the structure of cellulose and hemicellulose, which functions as a natural adhesive and provides strength and protection against degradation. Partial or complete removal of lignin is required to increase the accessibility of cellulose and hemicellulose and reduce bottlenecks in chemical or fermentation processes. When delignification occurs, the lignin is removed from biomass, and various changes occur in cellulose and hemicellulose. Lignin has strong bonds with cellulose and hemicellulose, which can hinder their separation. The delignification process breaks these bonds, separates the lignin from the cellulose and hemicellulose, and allows further processing. Delignification was carried out using a basic sodium hydroxide (NaOH) solution. When NaOH solution is applied to lignin-containing biomass, chemical reactions and structural changes occur, leading to the lignin's partial or complete destruction. The bonds formed between lignin and other lignocellulosic compounds, such as hemicellulose and cellulose, are highly susceptible to alkaline degradation. The hydroxide ion will attack the C=O bond and form a tetrahedral compound. The intermediate substance is unstable and immediately rebounds with the double bond. The bond between lignin and cellulose is broken and replaced by hydroxide ions [11]. Cellulose is formed from long polymer chains comprising 15,000 monomers and connected by β-1,4 glycosidic bonds between the monomers. Between the monomers form hydrogen bonds, so cellulose is not readily degraded chemically or mechanically in ordinary organic solvents [2,12].

Although research to extract fiber from durian peel waste has been carried out, not much research has focused on the characterization of durian peel fiber for textile applications. The research focuses on determining the optimal cellulose fiber extraction method to produce natural fibers that meet fiber parameter standards as textile raw materials.

The research used durian peel waste as a raw material and was extracted by synergizing with mechanical pretreatment. Fibers were characterized to determine the fibers' morphology, fineness (micronaire), and tensile strength. In addition, the process conditions (ratio of biomass to solvent) effect on the fiber characteristics obtained were also studied. Fiber content analysis was carried out using the Chesson-Datta method, while the test parameters were carried out according to the existing standard procedures.

2 Methodology

2.1 Materials

The material used in the delignification process is the inside part of the local durian peel waste, which is white. Durian peel wastes were taken from Rumah Durian Cemara, Mulyosari, Surabaya. Distilled water was obtained from the laboratory. NaOH and H_2O_2 (Merck, Germany). NaOH solution had a concentration of 35% (v/ v). The Chesson-Datta method was used to analyze the chemical content of the fiber, and the chemicals needed included H_2SO_4 (Merck, Germany), with a concentration of 1 N, and distilled water.

2.2 Equipment

The research used a 1 L glass reactor with an oil bath to maintain the process temperature. The delignification process carried out at atmospheric pressure. Fig. 1 illustrate the delignification equipment of durian peel waste.

Fig. 1. Durian peel waste delignification equipment to produce natural fibers as textile raw materials

2.3 Procedure

Preliminary Treatment. The inside part of the durian peel waste, which is white, is separated from the pericarp and boiled for a particular time to make it becomes soft. After boiling, it was hit with a hammer slowly to avoid excessive cellulose and lignin decomposition. The treatment is continued until the durian peel waste becomes flat. After that, it is followed by washing to remove the gum and then drying.

Delignification of durian peel waste. Dried durian peel waste was put into a glass reactor at various biomass ratios: NaOH solution (1:40, 1:50, and 1:60 w/v). The delignification process was carried out at 90°C-100°C for a particular time in the presence of low-speed stirring. After the delignification, the heating and stirrer are turned off, and the fibers obtained are taken and washed until the wastewater is clear.

Bleaching process. The delignified fibers were bleached in a glass reactor containing 35% hydrogen peroxide (H_2O_2) for a particular time at 90-100°C with stirring. The fiber resulting from the bleaching process is then washed with water until clean and dried.

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Fiber characterization.

Analysis of the chemical composition of the fiber. The Chesson-Datta method was used to determine the chemical composition of durian peel fiber. Some samples are expressed as notation (a), added to 150 mL of distilled water, then refluxed for a particular time at a temperature of 90-100 °C. After reflux, the sample is filtered, washed with distilled water, and dried to a constant weight to obtain the residue. The residue is weighed and expressed by the notation (b). The residue (b) is mixed with 150 mL of 1 N H_2SO_4 and then refluxed for a particular time at a temperature of 90-100 °C. The sample is washed, dried, weighed, and expressed by the notation (c). The dried residue (c) was soaked in 72% H2SO4 at room temperature for 4 hours, covered with aluminum foil, then added to the bath with 1 N H_2SO_4 and refluxed for a specific time. Solids are washed, weighed, and expressed by the notation (d). Solid (d) is burned to ashes, weighed, and expressed by the notation (e). The chemical composition of the fiber is calculated as follows:

$$\text{Percentage of hemicellulose (\%)} = (b - c)/(b - e) \times 100\% \quad (1)$$

$$\text{Percentage of cellulose (\%)} = (c\text{-}d)/(b\text{-}e) \times 100\ \% \quad (2)$$

$$\text{Percentage of lignin (\%)} = (d - e)/(b\text{-}e) \times 100\ \% \quad (3)$$

Analysis of Fiber Morphology using Scanning Electron Microscopy (SEM). SEM analysis was carried out to determine the fiber's length and diameter dimensions. The fiber analysis of the length and diameter was carried out with magnifications of 100 x and 300 x

Tensile and elongation test of fiber. The tensile analysis is carried out to measure the fiber strength. The fiber strength was measured by applying the maximum force that the fiber to withstand tensile or strain forces before failure or cracking occurs [13]. The resulting fiber was also tested for its elongation. The elongation value measures the percentage of how much the length of a fiber changes before it fails or cracks when subjected to a tensile force. Elongation measures the elasticity and ductility of fiber, namely how much a material can stretch before it finally breaks.

Micronaire analysis. Micronaire analysis was performed to measure the fineness of the fibers obtained. The micronaire value is expressed in micrograms per 1 in, the average weight obtained for each length. A low micronaire value indicates that the fiber has high fineness, while a high micronaire value indicates a rough texture [13].

3. Results and Discussions

The research focused on knowing the ratio of the mass of durian peel waste and the solvent used in the delignification process on the characteristics of the fiber produced for textile applications. The fiber characteristics studied included chemical characteristics, dimensions (length and diameter), strength/stretchability, and fiber fineness. The process steps involved include mechanical pretreatment to obtain dry crude fiber from durian peel waste and proceed with the delignification process. The delignification process was carried out using an alkaline solvent.

3.1 Yield and Composition of Durian Peel Fiber Results from the Delignification Process

The delignification process uses a glass reactor with a stirrer and heater to maintain the system temperature. Stirring was carried out at 70-80 rpm, and the temperature was maintained at 90°C-100°C. Durian peel fiber was delignified using a ratio of crude fiber weight to alkaline solvent volume in the range of 1:40 w/v, 1:50 w/v,and 1:60 w/v. After the delignification process, the fiber washing process is carried out until it is clean, followed by the bleaching process.

Table 1 shows the fiber yield as an effect of the ratio of the weight of crude fiber to the alkaline solution volume.

Table 1. The yield of the fiber after the delignification process at various weight ratios of crude fiber to alkaline solvents

Ratio of Crude Fiber Weight to solvent, % w/v	% Yield
1: 40	56.49
1: 50	49.48
1: 60	54.74

Table 1 shows that the % yield obtained after the delignification process ranges from 49.48 to 56.49%. Based on the analysis of the initial composition of durian rind fiber, the composition includes hemicellulose 20.77%, cellulose 66.08%, and lignin as much as 13.15%. Delignification can significantly affect the yield of fiber obtained from lignocellulosic biomass. Fiber yield refers to the amount of cellulose fiber successfully extracted from biomass after undergoing certain separation and processing processes. The lignin and hemicellulose fractions are partially dissolved in an alkaline solution. Lignin contains various types of ester and ether chemical bonds that hold its phenolic units together. NaOH solution is alkaline and can break these ester and ether bonds through hydrolysis. The reaction results in lignin fragments that are smaller and

more soluble in alkaline solutions. In addition, the reaction can also produce phenolics and phenolic derivatives which are more soluble in water. Lignin has a hard and strong properties, which can make cellulose and hemicellulose difficult to access. When lignin is removed, the accessibility of cellulose and hemicellulose to chemical reagents improves. With the removal of lignin, the reactivity of hemicellulose increases, facilitating chemical reactions such as hydrolysis. It causes the yield decrease because the biomass loses the lignin and some hemicellulose structure. It is appropriate with research from [14], which states that lignin and hemicellulose fractions in corn cobs are partially dissolved in a dilute Alkaline solution (0 – 32 g NaOH in 100 g biomass) at 100-150 °C temperature within 1 – 60 minutes. Cellulose did not change significantly. These results are consistent with other studies that have been reported where the solubility of the lignin and hemicellulose fractions occurred at low base concentrations and high temperatures. Atthe same time, cellulose remained largely unaffected [15]. Meanwhile, according to [16], the hemicellulose fraction will undergo partial hydrolyzes, the bond between lignin and cellulose will be broken, and the lignin will dissolve in a concentrated alkaline solution. From the results obtained, the % yield is relativelysensitive to changes in the amount of alkaline solution used. The fiber yield obtained after the delignification process can be seen in Figure 1.

(a) (b) (c)

Fig. 2. Durian peel fiber after the delignification process with a ratio of crude fiber: NaOH solution: (a) 1:40 b/v; (b) 1:50 b/v; (c) 1:60 b/v

Fig 2 illustrates that the fibers obtained generally tend to be lumpy, have a rough texture, and are brown. However, there is a difference between the three-fiber produced, where fiber with a ratio of 1: 60 (w/v) (c) gives a more open and separable structure compared to fibers with a lower ratio of fiber and solvent (a and b). In addition, in terms of texture, fiber (c) is thinner and brighter in color than the other results. It occurred because the amount of NaOH in the variable 1: 60 w/v was the largest, so the fiber delignification process ran well. The more NaOH is used, the more lignin is removed from the biomass. This can change the proportion of cellulose and hemicellulose in the fiber. In addition, higher concentrations of NaOH can cause changes in the space between the fibers, affecting fiber density. Fibers with high cellulose content will be finer than fibers that still contain hemicellulose or lignin. Lignin is a protective compound for cellulose which makes fibers containing lignin tend to be coarser.

After the fiber is obtained from the delignification process, then washing and bleaching uses hydrogen peroxide. Hydrogen peroxide (H_2O_2) is a strong oxidative bleaching agent. When applied to the fiber, hydrogen peroxide interacts with the pigment components (phenolic compounds), natural dyes, and lignin residue in the fiber. Hydrogen peroxide releases active oxygen in the form of hydroxyl radicals (OH•), which are strong oxidizing agents. The hydroxyl radicals produced from hydrogen peroxide help oxidize the pigments and natural dyes in fibers. The oxidation process changes the chemical structure of pigments and dyes, turning them into compounds that are more soluble or more easily removed during the washing process. Hydrogen peroxide also interacts with residual lignin that may still be present in the fiber after the initial delignification stage. This results in bleaching of the fiber and reduces the overall color of the fiber.

The fiber content of the durian peel was analyzed using the Chesson-Datta method, as shown in Figure 2.

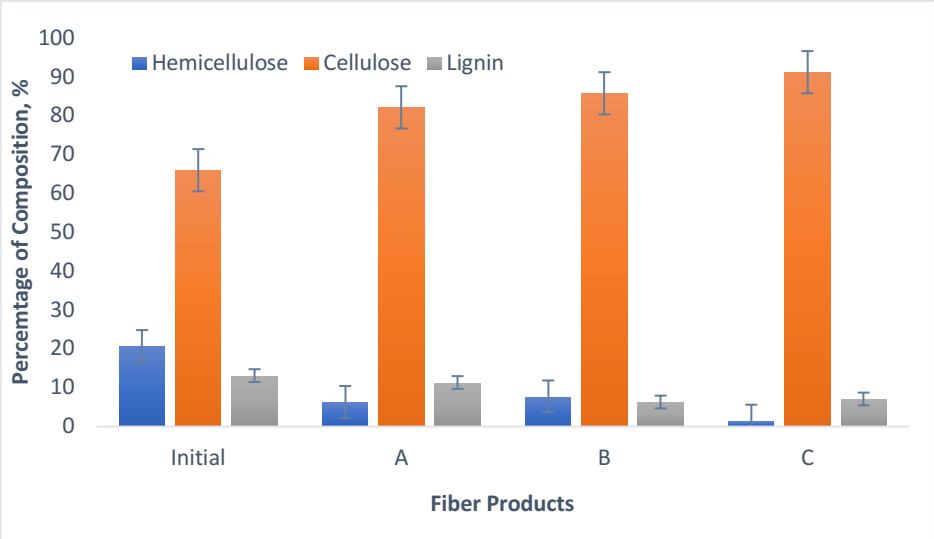

Fig. 3. Composition of durian rind fiber after delignification and bleaching process by Chesson - Datta analysis

Fig. 3 shows that the hemicellulose and lignin content in the fiber at various fiber-to-solvent ratios was lower when the amount of NaOH solution added increased. It follows the previous discussion that the partial hydrolysis of hemicellulose and lignin dissolution will occur during the delignification process using an alkaline solution. Lignin can function as physical and chemical barriers that block access to cellulose fibers in the biomass. In the delignification process, the partial or complete removal of lignin can improve the accessibility of cellulose fibers, making them easier to extract. Removing some of the lignin increases the proportion of cellulose fibers in the biomass relative to lignin and other components. It means that when lignocellulosic materials are processed to produce cellulose fibers, the fiber yields tend to be higher because more fibers are accessible. The hemicellulose composition of the delignified fibers, as shown in Figure 3, decreased significantly compared to the hemicellulose composition

of the initial durian peel fiber, which was 20.77 ± 1.3311%. The hemicellulose composition after processing with a ratio of 1:40 (A), 1:50 (B), and 1:60 (C), respectively, were 6.35%; 7.7854%; and 1.5374%.

The delignification process with a ratio of 1: 60 removed the hemicellulose content of up to ± 19% of the initial durian peel fiber. Hemicellulose is easier to undergo partial hydrolysis compared to other compounds because it has the weakest bond compared to cellulose and lignin, so the decrease in the initial durian peel fiber composition is quite significant. The lignin content was also seen to decrease when the amount of NaOH solution was increased. The lignin content in the initial durian peel fiber was 13.1492%, while the fiber that had been processed with different amounts of solvent was 11.3666% (A); on B of 6.3154%; and at C of 7.1209%. The maximum amount of lignin that can be removed reaches ± 6% of the lignin content in the initial durian skin fiber. It shows that the delignification process can destroy the lignin structure in the durian peel fiber and dissolve it. On another aspect, the cellulose content increased in the delignified durian rind fiber. The composition of the initial cellulose in the durian peel fiber reached 66.0780%. After being delignified with an increasing amount of solvent, it will increase the amount of cellulose in the fiber, successively the cellulose content of 82.2834% (A), B of 85.8992%, and the cellulose content of durian skin fiber C of 91.3417%. It indicates that the cellulose content obtained in the delignification process using a high amount of NaOH solution will also give a high cellulose content. The increasing amount of NaOH solvent will increase the lignin-dissolving process in the solvent. The more alkaline compounds in the system will increase the contact possibility between the alkaline compounds and cellulose.

3.2. Dimensions (Length and Diameter) of Durian Peel Fiber Delignification Process Results

Fiber dimensions (length and diameter) were analyzed using a Scanning Electron Microscope (SEM). The analysis results are shown in Figure 4. Fig. 4 shows that the durian peel fiber consists of branched fibers, not a single fiber, and is short in size.

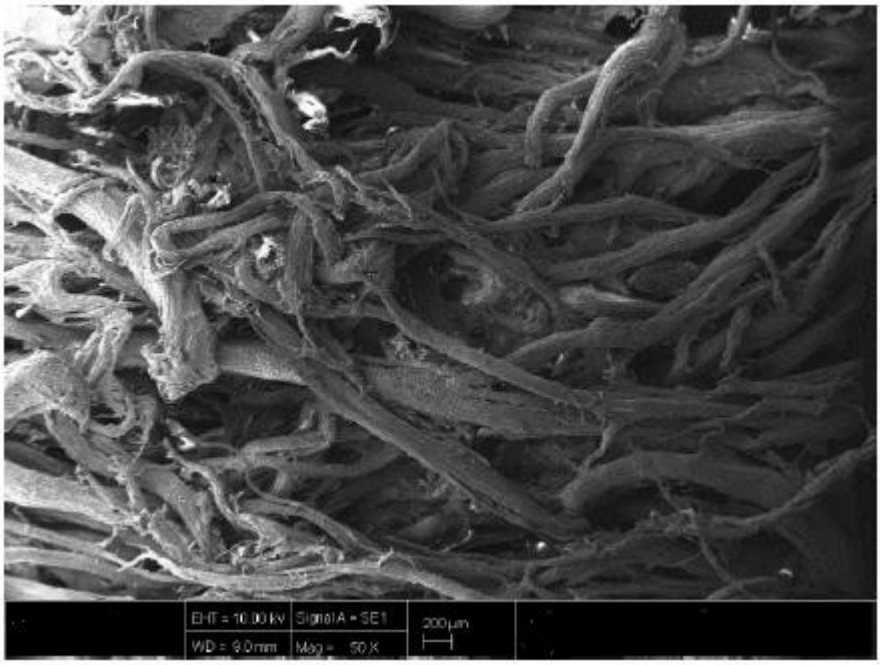

Fig. 4. Morphology of durian peel fiber with 50x magnification

Fig. 5 shows the fiber morphology at various amounts of NaOH solvent in the delignification process with a magnification of 300 times. In Fig. 5, it can be seen that there are long and uneven fiber lines; these lines indicate the presence of cellulose, lignin, and hemicellulose [17,18]. The composition reduction of lignin and hemicellulose reduces the diameter of the durian peel fiber. The size and shape of the durian peel fiber can change after the delignification process. An effective delignification process can reduce the lignin content in the fiber, making the fiber appear thinner and smoother. The lignin provides most of the thickness and stiffness of the fiber. The size reduction occurs because lignin and hemicellulose are removed in the primary cell wall of the fiber through delignification and bleaching processes (10), increasing the L/D ratio of the fiber. Therefore, the L/D ratio obtained increases with the loss of lignin and hemicellulose compounds.

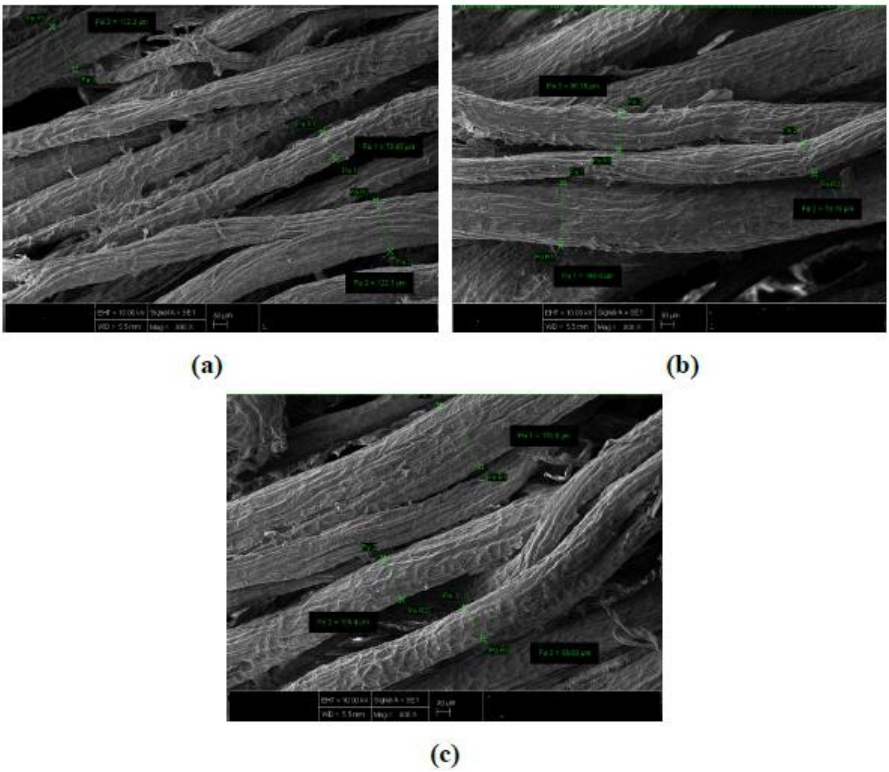

Fig. 5. Morphology of durian peel fiber at various fiber mass ratios and the amount of solvent at a magnification of 300 times (a) 1:40 %w/v; (b) 1:50 %b/v and (c) 1:60 %b/v

The length and diameter of the durian rind fiber were measured using an image from the Scanning Electron Microscope with a magnification of 300 times. The measurement results can be seen in Table 2. Table 2, it can be seen a comparison of some dimensional values of both research and commercial fiber textile fibers. The fiber L/D ratio of 1:100 is used as a textile fiber [19]. However, if the fiber is to be used as clothing fiber, then the ratio of the length and diameter of the fiber must be greater than 1:1000 [20]. It aims to obtain the flexibility properties of the fiber, which will facilitate the spinning process into yarn. Because the results of durian peel fiber obtained an L/D value of around 440, its durian peel fiber meets the minimum standards for the textile fiber's length and diameter requirements. However, it still needs to meet the standards for clothing fibers.

Thus, durian peel fiber cannot be used as clothing fiber but still fulfills the requirements as a textile fiber.

Table 2. The results of dimensions of durian skin fiber and other fibers with SEM

Type of Fiber	Fiber Length (mm)	Fiber Diameter (μm)	L/D
Durian peel	48	109	440
Cotton	25	17,5	17
Wool	75	25	3000
Silk	50.10^4	15	33.10^6
Hemp	150	50	3000
Jute	25	20	1250
Flax	25	15	1667
Sisal	3	24	125

3.3. Durian peel fiber strength, elongation, and smoothness resulting from the delignification process

Fibers used in the textile industry must have sufficient strength to avoid breaking when processed. For cotton fiber, the elongation ranges from 4-13%, with an average value of 7%. The elongation value of the fiber used for the textile industry is generally 10% of the length of the fiber itself [19]. Durian peel fiber has an elongation value of 1.285-3.142%, with an average value of 1.91%. The elongation value obtained from the stretch test is still lower than that of cotton fiber, which has an elongation value of 5-7%. The strength value of durian peel fiber has an average value of 0.56 g/den, which is still lower than textile fibers in general, such as cotton, which is standard with a strength value of 3-5 g/den [20]. The tests focused more on the fineness of the resulting fiber, where the average value of the fineness of the durian skin fiber produced was 34.5 microns. The results show that the durian peel fiber has rough properties compared to the fiber fineness value for wool and cotton fibers. The cellulose in cotton fiber has a neater and more compact structure, which makes it finer and more pliable.
On the other hand, the cellulose in durian peel fibers might be more disordered, resulting in a coarser structure. Durian peel fiber tends to have a higher lignin content than cotton fiber. Lignin is a component that gives fiber strength and structure but can also make fibers coarser. In addition, the different hemicellulose content in the two types of fiber can also affect roughness. The fiber surface of durian peel can have a rougher microscopic structure or have a more uneven physical appearance compared to cotton fiber which often has a smoother surface. The comparison of the strength, elongation, and fineness of durian skin fiber resulting from the delignification process when compared to other natural fibers can be seen in Table 3

Table 3. Comparison of strength, elongation, and smoothness of durian rind fiber against other natural fibers

The type of natural fiber	Strength, g/den	Elongation, %	Fineness, micron
Kulit Durian	0,56	1,91	34,5
Kapas	3-5	5-7	2,8
Rami	6-8	4-5	-
Jute	2,7-6	0,8-2	-
Flax	5,5-6,5	2-3	-

4. Conclusions

The research results found that the ratio of biomass: solvent of 1:60 b/v gave the highest cellulose content. Under optimum conditions, the ratio of length and diameter (L/D) is 440, so it can be seen that the fiber from durian peel waste is short. However, this value still meets the requirements of a textile fiber. In the tensile strength test, a value of 0.565 g/den was obtained, which indicated that the fiber had low strength and did not meet the standard value. The fiber elongation value obtained was 1.91%, meaning that the fiber was not flexible, and the results did not meet the standard elongation value. The micronaire value of durian skin fiber is 34.5; this means that the fiber is coarse, and the value does not meet the standards used. From the fiber tests carried out,durian skin fiber cannot be used as clothing fiber, although it can still be used as a textile fiber material for bag, wallet, sandals, etc.

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