Effect of Steam Delignification and Bleaching Process on Pineapple Leaf Fiber as Textile Raw Material

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***Abstract*— Pineapple is a local commodity in Indonesia that is widely cultivated. The part of the pineapple plant that is mostly used is only the fruit part, while the other plant parts are discarded and become waste. Pineapple leaves contain high fiber content of cellulose, lignin, and hemicellulose, so it has very potential if used as an alternative textile raw material. The quality of pineapple leaf fiber can be improved by going through a delignification process using an alkaline solvent with the appropriate concentration and time. This research determine the effect of delignification of pineapple leaf fiber with a solution of Sodium Hydroxide (NaOH), Hydrogen Peroxide (H_2O_2) and water (H_2O) on tensile strength, lignin content and SEM Analysis. This research consists of six stages including delignification using NaOH and H_2O_2, washing, neutralization, and analysis of pineapple leaf fiber products. The results obtained are pineapple leaf fiber lignin test after delignification and bleaching, the best lignin results are steam delignification at 80°C with 3% NaOH solution and bleaching solution using NaOCl solution obtained 21% lignin content with a tensile strength test of 0,263 kgf/mm^2. SEM analysis also showed that there was a loss of material from the fiber surface indicated there was degradation.**

***Keywords*— Alkaline, Bleaching, Pineapple leaf, Steam delignification**

I. INTRODUCTION

Pineapple (*Ananas comosus L.*) is one of the superior fruits in Indonesia. This is supported by the large production of pineapple which is in the third position after bananas and mangoes. Pineapple plants in Indonesia are very developed and spread in several areas, pineapple plantations in Indonesia are found in Lampung, Yogyakarta, Bogor, Central Java, East Java, and several other areas.

In Indonesia, pineapples are often found and cultivated. Pineapple plants need to be replanted if they have been harvested for two or three times. So far, pineapple plants are discarded at harvest only as organic waste, due to lack of knowledge about the potential and processing of Pineapple Leaf Fiber (PALF). Pineapple leaf fiber can be used as agricultural and non-food products that have developed for a long time, especially in the textile industry. Based on its physical and mechanical properties, natural fibers have high tensile strength and Young's modulus, good thermal and electrical resistance. Based on its chemical properties, the fiber has a high cellulose content, which has a relationship with tensile strength, crystallinity, and density. However, natural fibers also have disadvantages such as poor moisture resistance and low adhesion efficiency [1].

TABLE 1.
COMPARISON OF LIGNIN CONTENT IN NATURAL FIBERS [2]

Natural Fiber	Lignin Content (%)
Pineapple	4.6-12
Ramie	0.6-1
Cotton	0.5-1

Based on Table 1 that pineapple leaf fiber has a low lignin content, delignification was carried out to separate the cellulose adhesive contained in the fiber which has rigid and brittle properties. Lignin can affect the formation of bonds between fibers and can reduce whiteness [3]. The purpose of the delignification treatment is to change the physical properties of PALF by removing the lignin cementing layer in the fiber, fiber processing is carried out to separate the technical fiber bundles into smaller units and turn them into softer, therefore, mechanical properties such as ductility (g/tex) and the average peak elongation can be increased following the surface smoothness. There are other biological methods using enzymes, however, the enzymatic treatment studied was less carried out than the alkaline treatment in removing the lignin content other than hemicellulose and wax [4].

The research of Trache et al. (2017) the process after delignification, bleaching which can be carried out using NaOCl solution with stirring at a temperature of 75°C for 1 hour to remove residual protein and some chromomobility, this process can be carried out repeatedly until the fiber produces a white color [5].

Based on research conducted by Gaba et al. (2021) Alkali Treatment was performed with NaOH concentration of 1, 3, 6, 9% and soaked for 1 hour [6]. Then research by Gnanasekaran et al. (2020), conducted steam treatment of pineapple fibers, the fiber was evaporated at a temperature of 121°C with a pressure of 21 psi for 1 hour using an Autoclave. The bleaching process used 1.7% $NaClO_2$ concentration and mixed 0.2 M CH_3COOH at 80°C for 4 hours, with a ratio of 20:1. After that, it was neutralized using H_2O to pH 7-8 and dried for 1 day [7]. The results of the research above, it was found

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that the greatest tensile strength value was produced in pineapple fiber which was treated with alkaline treatment with a NaOH concentration of 6%, which was 1620 MPa [8].

This research is a development of previous research conducted by Triastuti, W.E (2021) where pineapple leaf fiber was delignified using NaOH with a concentration of 2-7% for 2 hours at 80oC by soaking. The results showed that the fibers that produced the best tensile strength were those that were delignified at a NaOH concentration of 6% [9].

From the studies above, NaOH solvent is mostly used for the delignification process of natural fibers. In the above research, delignification using the steam method was also carried out and there was also research with bleaching treatment. Steam delignification is often used as a pre-treatment before chemical bleaching. By softening and loosening the lignin, it prepares the fibers for more effective lignin removal during the subsequent bleaching steps. Therefore, in this study, delignification was carried out using NaOH solvent with variations in the concentration of the solvent. Delignification is carried out using the steam method with variations in steam temperature and further treatment is also carried out, namely bleaching with various types of bleaching solvents. This controlled approach helps to preserve the fiber's mechanical properties while still achieving effective lignin removal, resulting in fibers with improved properties for various applications.

II. METHOD

The research consists of six stages including pineapple leaf fiber extraction, delignification, washing, neutralization, preservation, and fiber analyzed. The materials used were pineapple leaves from the pineapple queen type (*Ananas comosus L.*) from Kediri, H_2O_2, H_2O, and NaOCl, NaOH and H_2SO_4 from Merck, and CH_3COOH.

The equipment used includes decorticators, autoclaves, and electric heaters. The variables used in this study were 30 minutes for the delignification time, 90 minutes for the bleaching time, the concentration of H_2O_2 and NaOCl of 3%, the concentration of NaOH 1; 2; 3; 4; and 5%, and delignification temperatures of 80°C and 120°C.

The first procedure carried out is the preparation stage, by preparing the main raw material, pineapple leaves by removing the flesh of the pineapple leaf skin until white fibers are visible and then taking the fibers. To take this fiber, use a decorticator machine by inserting pineapple leaves into the tool so that the pineapple leaf fibers will peel off from the skin of the pineapple leaves because of the cylinder with a knife blade in the machine. Then make a solution of NaOH and H_2O_2 with a predetermined concentration. The next step is delignification using steam with a variable solution of NaOH, H_2O_2, and H_2O at 80°C and 120°C which is the stage of contacting pineapple leaf fiber with steam. Then the washing stage where the pineapple leaf fiber is cleaned of residual solvents and impurities on the fiber by rinsing with water. The next step is neutralization by boiling the pineapple fiber at 100°C for 2 hours so that the remaining NaOH and H_2O_2 can be dissolved in water. Then the preservation stage is by drying the delignified pineapple leaf fiber, which is free from NaOH, H_2O_2, and H_2O until a constant weight is obtained. The fiber test phase was carried out with three tests, including lignin content, which is conducted by using Klakson method. The Klakson method was carried out using H_2SO_4 and soaked for 2 hours, then soaked again by adding H_2O, then heated for 4 hours. The lignin precipitate will settle, will be dried in an oven at a temperature of 105°C. SEM is conducted using an electron microscope that can display an image of the surface of a specimen with high resolution to determine the pores in the pineapple leaf fiber after the delignification process. Tensile strength analysis is a test by applying a force or stress to the material to determine or detect the strength of a material, the test conducted by pulling with a continuous tensile force so that the elongation of the material will continue to increase until it breaks.

III. RESULTS AND DISCUSSION

A. Analysis of the Effect of Delignification and Bleaching on Pineapple Leaf Fiber on Lignin Contents

The lignin test aims to determine the lignin content in pineapple leaf fiber after delignification and bleaching treatment. The Klason method was chosen to determine the lignin content in this study, the Klason method was carried out using 72% H_2SO_4 and soaked for 2 hours, then soaked again by adding H_2O up to 575 mL so that the H_2SO_4 concentration became 3%, then heated for 4 hours. The lignin precipitate will settle, and will be filtered and the lignin precipitate will be dried in an oven at a temperature of 105°C. Steam delignification on its own might have a limited impact on the tensile strength of pineapple fiber. It can potentially soften the lignin and make it more susceptible to removal in subsequent bleaching steps. However, since steam delignification is not as aggressive as chemical bleaching, it may have a minimal effect on the fiber's mechanical properties, and the tensile strength may not be significantly altered.

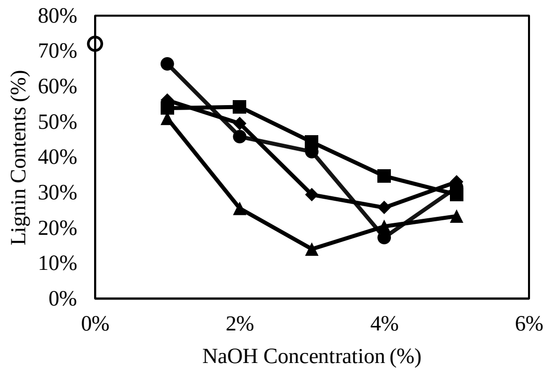


Figure 1. Effect of delignification and bleaching on pineapple leaf fiber on lignin contents. Note: ▲= H_2O_2 120ºC, ♦= H_2O_2 80ºC, ●= NaOCl 120ºC, ■= NaOCl 80ºC, ○= without treatment

From the research results listed, the NaOH concentration for delignification with a variable of 1-5%, the optimum concentration was produced at a variable concentration of 3% NaOH. Figure 1 show that the contents of lignin with delignification and bleaching variables tend to decrease with increasing NaOH concentration. The test results above show that the lowest lignin content is produced at a steam temperature of 120°C, with a lignin content of 14% with a tensile test value shown in Figure 1 of 0.239 kgf/mm^2. Other quite good results were also produced in 4% NaOH solution at a temperature of 80°C with a bleaching solution of NaOCl with a lignin content of 21% in terms of the tensile test value in Figure 1, which is quite high, namely 0.263 kgf/mm^2. This happens because the high temperature affects the depolymerized lignin. The increase in temperature causes thermal softening of the lignin polymer, causing an increase in lignin depolymerization in an acidic or alkaline environment [10]. Steam treatment also affects the resulting delignification results, according to Prosvirnikov, et al (2017) active lignocellulosic materials, react faster at higher temperatures and steam treatment makes a state of high humidity facilitating accelerated saturation of the liquid into the interior. particles, then the lignin destruction reaction starts first about 1.5 - 2 times faster [11].

Based on the results of the study above, it also showed that treatment using H_2O_2 solution produced lower lignin content than NaOCl solution. Solvent bleaching using NaOCl and H_2O_2 resulted in a large content of cellulose where the content of lignin remaining in the fiber was small. The H_2O_2 solvent produces cellulose, which is larger than NaOCl, this is because NaOCl produces HOCl which is acidic so that it can dissolve cellulose. The bleaching method using these two solvents can change the color and reduce the lignin content so that the resulting product has a brighter color [12]. NaOCl is a powerful oxidizing agent and acts as a strong bleach. It can effectively break down and remove lignin residues that might not have been completely removed by other treatments, including NaOH. The oxidizing action of NaOCl helps to further degrade and solubilize lignin, leading to additional lignin removal from the fiber.

B. *Analysis of the Effect of Delignification and Bleaching on Pineapple Leaf Fiber on Tensile Strength Test*

A tensile strength test was carried out to determine the tensile strength of pineapple leaf fiber after the delignification process. Tensile test is a test conducted by applying a force or stress to the material to determine or detect the strength of a material. The tensile strength test of a material is carried out by pulling with a continuous tensile force so that the elongation of the material will continue to increase until it breaks.

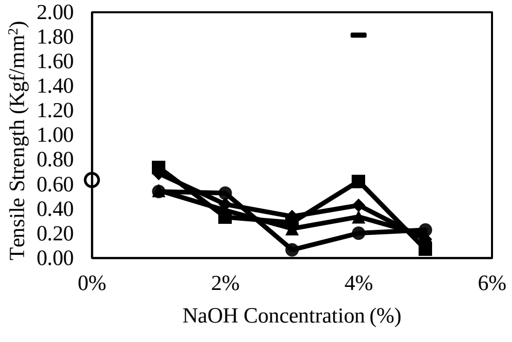
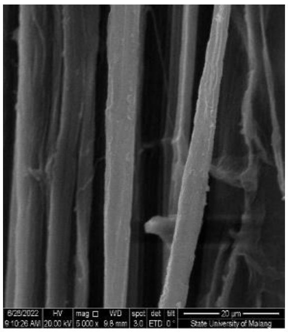


Figure 2. Effect of delignification and bleaching on pineapple leaf fiber on tensile strength test. Note: ▲= H_2O_2 120ºC, ♦= H_2O_2 80ºC, ●= NaOCl 120ºC, ■= NaOCl 80ºC, ○= without treatment, - = Fiber

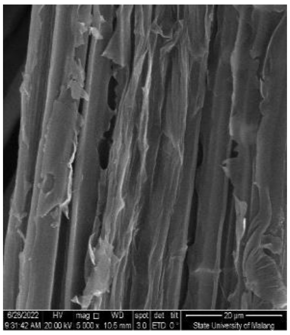
Based on Figure 2, the treated fiber experienced a decrease in the tensile strength test compared to the untreated fiber, and as the concentration of NaOH used increased, the tensile strength test results also decreased. The data from figure 2 shows a decrease in fiber tensile strength test results about 4-89%. While bleaching treatment has an impact in increasing the fineness up to 5-6%, bleaching can reduce tensile strength up to 40-45% [8]. Based on the results of the study, there was a decrease in fiber tensile strength at an increasing NaOH concentration, this was due to the loss of hemicellulose, lignin, and pectin content so that the fiber strength decreased because the collection of fiber-forming microfibrils held together by lignin and pectin would be separated so that the amount of tension that could be achieved withholding is reduced [12]. Meanwhile, in a study conducted by Wang et al. (2018) delignified fibers can form hydrogen bridges from the OH group which causes the fiber to increase its tensile strength [13]. NaOCl, if used at high concentrations or for prolonged periods, can lead to fiber damage and a decrease in tensile strength. However, when properly controlled and used at appropriate concentrations, NaOCl can still be effective in removing lignin while minimizing damage to the cellulose structure. Careful optimization of the bleaching conditions helps preserve the fiber's tensile strength to an acceptable level. while there is generally a positive correlation between delignification and tensile strength during the initial stages, it is essential to find the optimal point of delignification to achieve the best mechanical properties in the fiber. Over delignification should be avoided to prevent a decline in tensile strength. The correlation is influenced by various factors, and a careful balance must be maintained during the delignification process to achieve the desired mechanical performance in the treated fibers.

C. *Analysis of the Effect of Delignification and Bleaching on Pineapple Leaf Fiber on the Scanning Electron Microscope (SEM) Test*

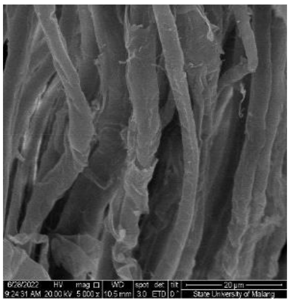
SEM test is performed to observe the morphology of a material. SEM can be used to examine the surface morphology of the pineapple fiber before and after bleaching. Successful bleaching would remove impurities and substances from the fiber's surface, resulting in cleaner and smoother fibers. The SEM test aims to determine the pores in the pineapple leaf fiber after the delignification process. SEM is an electron microscope that can display an image of the surface of a specimen with high resolution. The SEM test was carried out on untreated fibers and the best variable was the results of the lignin test and fiber tensile strength test, namely the 4% NaOH delignification solution variable at 80°C with NaOCl bleaching treatment and other variables as comparison, namely 4% NaOH delignification at 120°C with H_2O_2 bleaching treatment. Steam delignification can lead to changes in the fiber's microstructure, including alterations in the arrangement and distribution of fibers and other constituents. These structural changes may be visible in SEM images, providing insights into the effects of delignification on the fiber's morphology.



(a)



(b)



(c)

Figure 3. Fiber morphology results (a) no treatment, (b) NaOCl 80°C, and (c) H_2O_2 120°C

Based on Figure 3, it shows that there are morphological differences in the fiber of pineapple leaves that are treated and vice versa. In the fiber that was not given any treatment (a) no peeling occurred with a lignin content of 72%. Meanwhile, the fibers which were given delignification treatment using 4% NaOH solution and bleaching using NaOCl solution with a steam temperature of 80°C (a) produced a lignin content of 21% and a 3% H_2O_2 solution with a steam temperature of 120°C (c) resulted in a lignin content of 21%. 20% showed that there was peeling of the fiber layer caused by a decrease in lignin content of 71% and 72% from the delignification and bleaching treatment of NaOCl 80°C and H_2O_2 120°C. This shows that the loss of material from the fiber surface indicates that the degradation occurs due to the addition of bleaching solution. The contents of lignin H_2O_2 120°C are lower than NaOCl 80°C as shown in Figure 3 (c) the peeling occurs more significantly than Figure 3 (b) this is because the high temperature affects the depolymerized lignin. The increase in temperature causes thermal softening of the lignin polymer, causing an increase in lignin depolymerization in an acidic or alkaline environment [10]. Pulp treated with bleaching has a high level of brightness because the addition of bleaching increases the accessibility of chemicals in the form of bleach to the pulp and allows the removal of less degraded lignin [11]. Treatment with alkali can cause thinning or reduction in the structure of the fiber followed by the breaking of fiber bonds. Alkaline treatment can also cause discoloration of natural fibers due to the removal of natural dyes in natural fibers [14]. Steam delignification can soften and loosen the lignin present in the fiber's cell walls, potentially leading to a smoother fiber surface. The removal of lignin and other impurities can contribute to a cleaner and more even surface appearance in scanning electron microscopy (SEM) images.

IV. CONCLUSION

The results of the pineapple leaf fiber lignin test after steam delignification at a temperature of 80°C with 3% NaOH solution and bleaching solution using NaOCl of 21%o give the best value and tensile strength test is 0.263 kgf/mm^2. The SEM analysis showed that after treatment the fibers peeled off compared to the untreated fibers with a 71% decrease in lignin content. Yarn from pineapple leaf fiber can be used as a fabric with a tensile strength test value that has met the requirements of 17.84 cN/tex.

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