Effect of sonification in the alkalization process of coconut fiber to improve fiber strength

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Abstract. Coconut coir fiber is a natural fiber obtained from the coconut mesocarp, where in Indonesia, its utilization is still not optimal. The use of fiber as a functional material in the structural and filter requires optimal characteristics and related to lignocellulose content and modification of the fiber's surface structure. One method of fiber modification that is mostly used is the alkalization process using NaOH solution, which aims to degrade lignin, which is rigid and brittle. This paper will discuss the effect of ultrasonic wave exposure (sonication) on alkalizing the fiber using a low concentration NaOH solution. The variation of the concentrations of NaOH solution used was 1, 3, 5, 7, and 9% with ultrasonic wave exposure for 30 minutes at temperatures of 50, 60, and 70°C. The mechanical strength of the fiber was characterized using a Thwing-Albert Testing Machine. The measurement results show that the maximum mechanical strength values obtained in the alkalization treatment for 30 minutes with a concentration of 5% NaOH solution at a temperature of 70°C is 295.13 MPa. This tensile strength increased by 68.33% compared to the untreated sample of 175.33 MPa. The strain obtained in this sample was 31.86% of the initial fiber length.

1. Introduction

The use of natural fibers as a substitute for synthetic fibers began to be widely used by industries. This is because synthetic fibers have several disadvantages, such as limited resources, high production prices, and difficulty recycling. For example, Toyota Inc. in Japan already uses kenaf fiber for composite materials on car interior panels. The use of natural fibers is preferred because it can cover the weaknesses that exist in synthetic fibers [1].

Natural fibers are fibers that are directly obtained from plants. Natural fibers are generally divided into two groups, namely wood fiber and non-wood fiber. Non-wood fiber is divided based on the origin of plant parts, including [2]:
1. Fruit, for example: coconut (Nucifera).
2. Tree bark, for example: kenaf (Hibiscus Cannabius).
3. Leaves, for example: sisal (Agave Sisalana).
4. Grass fiber, for example: elephant grass (Eriathus Elephantinus).

One of the natural fibers that can be found in all corners of Indonesia is coconut coir fiber, which comes from the coconut mesocarp. Coconut production in Indonesia is 15.5 billion annually [3]. However, the utilization of coconut coir fiber is still not optimal, so that it is only used as a conventional handicraft product, whereas coconut coir fiber has a high mechanical property potential [4]. Coconut coir fiber has mechanical properties in the form of a stress of 175-220 MPa with a strain of 15-30% before being given treatment [5]. Giving treatment to increase coconut coir fiber can be done in order that coconut coir fiber can compete with other natural fibers to replace synthetic fibers.
Coconut coir fiber has three main components, namely cellulose (37.9%), hemicellulose (15.5%), and lignin (33.5%) [6]. Cellulose is a polymer composed of glucose monomers that gives the main mechanical strength to the fiber. Cellulose has a strong alkali resistance, is easily hydrolyzed by acids into water-soluble sugars, and heat resistance up to 211-280°C [2]. Hemicellulose is a matrix of cellulose, which is alkali resistant to a concentration of 18.5%. Lignin is a fiber content that has stiffness properties, thereby reducing the mechanical property of the fiber. This Lignin compound is an amorphous complex and has a high degree of polymerization. It is resistant to degradation both biologically and chemically but is easily dissolved in alkali [6]. In some materials that use natural fibers as components, lignin is often minimized or modified to obtain material that is not rigid and increases its mechanical strength. Lignin can be degraded by giving an alkali treatment [7].

Alkali treatment is one of the best treatments for strengthening natural fibers. A vital modification made by alkalization is to disrupt hydrogen bonds in structural networks and increase surface roughness. This treatment removes a certain amount of lignin, wax, and oil, covering the outer surface of the cell wall of fiber and depolymerizes cellulose. Adding liquid NaOH to natural fibers will increase the ionization of the hydroxyl group to an alkoxide. This alkalization reaction can be seen in the process, such as [8]:

\[ \text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - (\text{ONa}) + \text{H}_2\text{O} \]

It is feared that excessive use of chemicals can harm the environment. Therefore, the alkalization process will be assisted by exposure to ultrasonic waves to get more efficient treatment. The provision of ultrasonic waves in a solution will cause a cavitation effect, where the molecules in the solution oscillate to their average position and eventually cause a cave. The solution is stretched and dense. When the ultrasonic wave energy supplied is large enough, the wave strain can break molecular bonds between solutions. This causes the gases in the solution to be trapped due to the solution molecules whose bonds are broken when the dense occurs again [9]. This will have an impact on the decay of lignin as an effect of the acoustic cavitation.

2. Research Method
The research was carried out in the Material Physics Laboratory, Plasma Physics Laboratory, and Biophysics Laboratory of the Mathematics and Natural Sciences Faculty, Brawijaya University, from February to April 2020. The tools used in this study include ultrasonic cleaner, optical microscope, tensile test instruments, 100ml and 200ml beakers, spatulas, petri dishes, digital balance sheets, and plastic containers. Materials used include coconut coir fiber, NaOH crystals, and distilled water. The research parameters were divided based on concentration and temperature with 30 minutes of treatment duration as follows:
1. 1% NaOH concentration (T = 50°, 60°, 70°C)
2. 3% NaOH concentration (T = 50°, 60°, 70°C)
3. 5% NaOH concentration (T = 50°, 60°, 70°C)
4. 7% NaOH concentration (T = 50°, 60°, 70°C)
5. 9% NaOH concentration (T = 50°, 60°, 70°C)

2.1. Preparation
Coconut coir fibers are cleaned and weighed 12 grams for each treatment. Then determined the mass of NaOH for each concentration using the function of percent mass per volume of solute:

\[ \frac{\text{mass of solute}}{\text{mass of solvent}} \times 100\% \]

The mass of NaOH obtained was then dissolved in 100 ml distilled water
2.2. Treatment of Alkalization-Sonification
Coconut coir fibers are alkalized in NaOH solution aided by exposure to ultrasonic waves. The treatment was carried out in an ultrasonic cleaner with a frequency of 40KHz with a treatment duration of 30 minutes. Coconut coir fibers from the treatment then rinsed thoroughly and dried in the room temperature for 2x24 hours and roasted 120°C for 2 hours.

2.3 Testing
Coir fiber testing includes mechanical strength testing in the form of stress-strain. The test is carried out by giving a pair of opposing linear forces at both ends of the sample, coinciding on the rod's axis, and working through the center of the tensile test instrument's cross-section. Force intensity per unit area as a function of stress and change in sample length during testing as a strain function with the following equation:

\[
\sigma = \frac{F}{A} \\
\varepsilon = \frac{L_0 - L}{L_0} \\
\]

The standard used is the Japan Industrial Standard (JIS-R 7601) [2]. Fiber test samples should be formed in accordance with the standards, as in the figure 1 below:

![Figure 1. Test sample standards](image)

3. Result and Discussion

3.1. Tensile Stress Test
The tensile stress value of coconut coir fiber shows that the treatment of NaOH concentrations of 1%, 3%, 5%, and 7% have the same constructive results or are increasing the value of the tensile stress. The highest tensile stress value was obtained in the treatment of 5% NaOH concentration with a temperature of 70°C for 30 minutes, which is 295.13MPa or an increase of 68.33% of the coconut coir fiber without treatment. This indicates that treatment with these parameters can effectively degrade stiff lignin so that the presence of strength-giving cellulose becomes more dominant. The 9% NaOH concentration treatment has a harmful or damaging tensile stress value. The lowest tensile stress value was obtained in the treatment of 9% NaOH concentration with a temperature of 70°C for 30 minutes, namely 159.72MPa, or decreased by 8.90% of the fiber without treatment. This happens because the treatment with these parameters degrades lignin and degrades other components such as cellulose to damage the tensile stress value.
Table 1. Tensile stress test results of coconut coir fiber

| Concentration Alkalization (%) | T (°C) | Stress (MPa) |
|-------------------------------|--------|--------------|
| Without treatment -           | -      | 175.33       |
| 1                             | 50     | 189.11       |
|                               | 60     | 200.21       |
|                               | 70     | 192.82       |
| 3                             | 50     | 198.81       |
|                               | 60     | 186.18       |
|                               | 70     | 190.49       |
| 5                             | 50     | 237.88       |
|                               | 60     | 256.10       |
|                               | 70     | 295.13       |
| 7                             | 50     | 224.44       |
|                               | 60     | 224.69       |
|                               | 70     | 218.39       |
| 9                             | 50     | 191.64       |
|                               | 60     | 179.69       |
|                               | 70     | 159.72       |

Based on the stress test chart, it can be seen that an increase in NaOH concentration up to 5% will continue to increase the tensile stress value of the fiber. The stress value is maximal because, at the 7% and 9% levels, the value starts to decrease compared to the 5% content.

3.2. Tensile Strain Test

The value of coconut coir fiber tensile strain has a constructive value in the treatment of 5% and 7% NaOH concentrations with the highest strain value at a concentration of 5%, 70°C temperature of 31.86% of the fiber without treatment which is 24.84%. This indicates that the treatment with these parameters is also the best treatment to increase the coconut coir fiber strain, as well as the stress. The lowest strain value obtained at the treatment concentration of 9%, temperature of 70°C of 17.13%. The strain value decreases because the fiber content is excessively degraded, especially cellulose, which is degraded so that the mechanical strength value of the fiber is reduced. This means that the lower the strain value, the stiffer the fiber is.

Table 2. Tensile strain test results of coconut coir fiber

| Concentration Alkalization (%) | T (°C) | Strain (%) |
|--------------------------------|--------|------------|
| Without treatment -            | -      | 24.84      |
| 1                              | 50     | 24.85      |
|                                | 60     | 24.15      |
|                                | 70     | 23.61      |
3.3. **Observation of the Fiber Structure**

This research is aided by exposure to ultrasonic waves so that the alkalization process runs more efficiently. The fiber with the best test results is observed through an optical microscope with magnifications up to 1000x.

An increase in the coconut coir fiber's mechanical value is inseparable from the role of ultrasonic wave exposure. Ultrasonic waves that are propagated through the medium will cause the particles to vibrate with an amplitude parallel to the direction of propagation longitudinally, thereby causing the particles to oscillate to form a tight-density medium. As shown in figure 2, after experiencing strain tightness, medium particles cannot continuously hold the equilibrium point so that the saturation position will cause the particles to explode or collapse. This particle's explosion will degrade lignin located in the middle lamella directly, causing a cave on the fiber surface.

**Figure 2.** A fiber surface profile (a) before and (b) after treatment

4. **Conclusion**

Based on the results of this study, it can be concluded that the alkalization treatment using a NaOH solution with a concentration of 5% aided with ultrasonic wave exposure for 30 minutes at a temperature of 70°C can increase the tensile strength to 68.33% or by 295.13 MPa from the previous 175.33 MPa. The resulting strain also increased to 31.86% of the initial fiber length. The increase in tensile strength is caused by the degradation of lignin, which gives rigidity to the fiber so that the presence of cellulose, which gives strength to the coconut coir fiber, becomes more dominant.

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