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The long microfiber of Water-chestnut (*Eleocharis dulcis*) as composite reinforcing material

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Key words: Water chesnut (*Eleocharis dulcis*), Natural fiber, Alkalizing of 6% NaOH, Microfibers, Composite

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Abstract

This preliminary research used long microfiber from water chesnut (*Eleocharis dulcis*) which is a weed plant in South Kalimantan, as a composite reinforcement material. This study aimed to study the effect of 6% NaOH alkalizing treatment and ultra-sonication on the properties of a long microfiber of water chesnut, especially for their chemical, physical, and mechanical properties. Firstly, the fiber of water chesnut was alkalizing by NaOH. This 6% NaOH treatment has reduced the moisture content of the fibers and their chemical components (lignin, hemicellulose, and cellulose) while the density and tensile strength of the fibers were increased. The process continued by bleaching and ultra-sonication the fiber to create a long microfiber. The water content of fibers after alkalizing treatment and ultra-sonication has met the SNI standard. Alkalizing treatment reduced the fiber size by 373.05 μm, and in the form of microfiber, before ultra-sonication was 4.28-5.96 μm and after ultra-sonication was 3.17-4.00 μm. The tensile strength of long microfiber could not measure but after alkalized, it increased significantly, from 354.25 to 3,282.10 MPa. Therefore, it is recommended that this long water chesnut microfiber is very good when used as a reinforcing material in the manufacture of composites because it will blend better with the matrix.

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Introduction

Composites are a combination of two or more materials that have functions and uses as reinforcement and matrix. The use of natural fibers as composite reinforcement is an alternative material to replace metal materials. Natural fiber has several advantages such as: low density, biodegradable, and has good mechanical properties and most importantly environmentally friendly (Wang, 2003; Mallick, 2007). Natural fibers found in South Kalimantan are water chesnut (*Purun tikus*), bundung, bemban, water hyacinth, and others. Water chesnut or *Eleocharis dulcis* is a wild plant that can well adapt in waterlogged areas, both in tidal swamps, lowland swamps and rice fields. Water chesnut can grow in acidic soils with a pH of 2.5-3.5 (Rosyidah *et al.*, 2018). This plant has a fairly strong fiber because it has a rope-like fiber texture that can exceed the tensile strength of rattan or bamboo (Asikin and Thamrin, 2012; Firda and Fuad, 2020).

For many years, the water chesnut has been used by local community just for making mats (Asikin & Thamrin, 2012). Several studies have done for the possible potential of water chesnut such as biofilter (Prihatini *et al.*, 2011), heavy metal absorber (Asikin & Thamrin, 2012), activated carbon (Suryajaya *et al.*, 2020), and cement board composite material (Haryanti & Wardhana, 2017; Wardhana & Haryanti, 2017). Alkalizing treatment is the most common and best method in processing natural fibers to be used as a composite reinforcement material to improve the mechanical, physical and chemical properties of the fiber. The purpose of alkalizing is to break down the lignin structure, break down cellulose bonds, increase the porosity of the material, break down hemicellulose and depolymerize hemicellulose. From several studies that have been carried out, no one has used long fiber of water chesnut as a reinforcing material in the manufacture of composites. Besides that, research on the treatment of alkalization on long fibers of water chesnut to microfiber-shaped fibers has not been carried out. The size of the fiber affects the performance of the composite, as the diameter become smaller, the tensile strength would increase. It is caused by smaller voids in the fiber which in turn caused many intermolecular bonds, thus increasing its strength (Saidah *et al.*, 2018). It could be concluded that microfiber would be better in used than normal fiber. The natural fibers can be made into microfiber by isolating cellulose through the process of alkalization and ultra-sonication. In this article, alkalization and ultra-sonication treatments were carried out on natural fibers of water chesnut using long and straight fibers with a size of 20cm and this is the novelty of this study.

Ultrasonic treatment of cellulose could reduce porosity. Furthermore, it also increases fiber dispersion in the matrix and adhesion between matrix and fiber thus resulting in high tensile strength. In order to get a fine and strong microfiber dispersion, it is necessary to know the size of the microfiber and increase the number of hydroxyl groups on the surface of the fiber in order to increase the cellulose content (Kanoth *et al.*, 2017; Abral, Putra, Asrofi, Park & Kim, 2018; Chen *et al.*, 2011; Lismeri *et al.*, 2018). Therefore, the purpose of this study was to determine the characteristics (chemical, physical, mechanical), functional groups and chemical content of water chestnut fiber and to make microfibers with 6% NaOH alkalization for 3 hours and continued with the first bleaching process using 5% NaOCl for 2 hours, the second bleaching process using 4% KOH for 1 hour and continued by ultra-sonication process.

Materials and methods

Water chestnuts which have a length ranging from 100-160cm are cleaned and dried in the sun for 2 x 8 hours. To make the fiber, the dried chestnuts are cut 20cm long and split into 2 parts and then combed to get long and fine fibers. In previous studies, chestnut has a structure consisting of lignin, hemicellulose and cellulose where the cellulose in chestnut can be used as natural fiber for reinforcing material, so that lignin and hemicellulose which provide rigidity and protection to the fiber need to be removed (Haryanti *et al.*, 2021).

The process of making microfiber is done by chemical and physical treatment. The fiber refinement step was carried out with 6% NaOH alkali treatment with the aim of removing the less effective fiber constituent

components, which are hemicellulose, lignin, and pectin. Furthermore, the fiber is bleached twice. In the first bleaching process, 5% NaOCl was used for 2 hours; it was carried out as a more optimal dissolving process of lignin and hemicellulose.

Then the second bleaching process using 4% KOH for 1 hour is used for degrading lignin by hydrolysis. As result of the second bleaching process, the fibers turned to be smoother. This alkalization method would be carried out together with mechanical treatment by ultra-sonication for 1 hour at a temperature of 60°C to reduce the particle size of the water chestnut fiber into microfibers (Harpendi, Padil and Yelmida, 2014; Syafri *et al.*, 2018).

In a previous study, the microfiber was make from short fibers of water chestnut and the alkalization process using 5% NaOH, and 2% $KMnO_4$, while for bleaching process, 5% H_2O_2, and 50% H_2SO_4 was used (Haryanti *et al.*, 2021). In this study, instead of short fiber, the long fiber (20 cm) was used and alkalizing in 6% NaOH. While in the microfiber manufacturing process (bleaching), 5% NaOCl and 4% KOH was used for 2 hours and 1 hour, respectively. This is the novelty of this research.

To determine the feasibility of water chestnut fiber as a composite material by referring to the SNI 06-3730-1995 standard, the maximum moisture content must be less than 15% (Sahara *et al.*, 2017). The water content of water chestnut calculation is as follows:

$$\text{Moisture content (\%)} = \frac{A-B}{C} \times 100\% \quad (1)$$

Where, A= mass (sample + cup) before drying; B= mass (sample + cup) after drying; C= mass of the sample.

The lignin content was calculated as a percentage of lignin in water chestnut fiber, based on SNI 0492-1989-A, referring to equation (2):

$$\text{Lignin (\%)} = \frac{A}{B} x\ 100\% \quad (2)$$

Where, A= weight of lignin precipitate; B= dry sample weight Cellulose content was calculated as a percentage of cellulose in water chestnut fiber, based on SNI 14-0444-1989, referring to equation (3):

$$\text{Cellulose (\%)} = \frac{A}{B} x\ 100\% \quad (3)$$

Where, A= weight of cellulose precipitate; B= dry sample weight. The hemicellulose content was carried out according to SNI 14-0444-1989, referring to equation (4):

$$\text{Hemicellulose (\%)} = ((V_2 - V_1)\ x\ N\ x\ 6{,}85)/W \quad (4)$$

Where, V_1 is the need for $Na_2S_2O_3$ in the filtrate filtration; V_2 is the requirement for $Na_2S_2O_3$ in blank filtration; N= Normality $Na_2S_2O_3$ and W is the weight of dry ingredients that have been oven-dried (grams).

The density of water chestnut was calculated by the ratio of the mass and volume of the increase in water using an equation (5). The mass of each sample of water chestnut fiber was weighed and then put into water and the increase in water volume was measured.

$$\rho = \frac{M}{V} \quad (5)$$

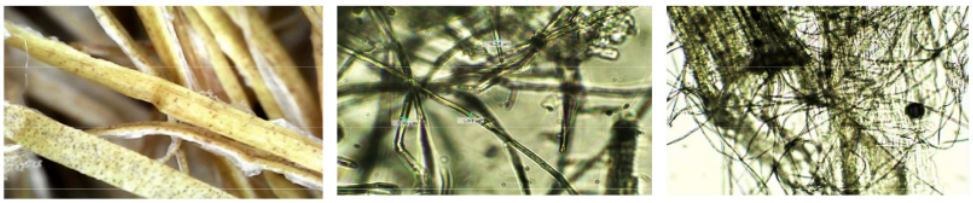
Where, M = fiber weight (grams); V = Volume added of water (ml)

Furthermore, FTIR was used to investigate the chemical content in water chestnut fiber. In addition, the tensile strength of water chestnut fiber was also tested. The fiber tensile strength test was carried out using a 20cm long fiber of water chestnut. The stages of analysis of the water chestnut tensile test were based on ASTM D3379-75 (Indonesian National Standard (SNI) No. 0444 -1989).

Result and discussions

The diameter of the water chestnut fiber was measured using a digital microscope and the ratio of the diameter of the sample is shown in Fig. 1. The fiber diameter was measured for 3 types of samples, namely fiber after 6% NaOH treatment, bleaching before ultra-sonication and after ultra-sonication. The size of the long fiber diameter of water chestnut after 6% NaOH treatment is shown in Fig. 1a, the diameter of the water chestnut fiber is 373.05 μm. These fibers appear coarse.

Meanwhile, after the fiber became microfiber and underwent a bleaching process of 5% NaOCl and 4% KOH but before ultra-sonication, the diameter of the fiber decreased significantly by 4.28-5.96 μm and looked like in Fig. 1b and after ultra-sonication the microfiber also decreased in diameter of 3.17-4.00 μm and looks like in Fig. 1c. Based on these results, the ultra-sonication process on water chestnut microfiber was better in fiber diameter than the 6% NaOH alkalization treatment. It also appears that the water chestnut microfiber before ultra-sonication looks coarser and the fiber diameter is larger than after the ultra-sonication or heating process where the fiber looks finer. This is caused by the reduction of lignin and hemicellulose (Fig. 1c). The diameter size of the water chestnut microfiber in this study was also better than that of the water chestnut short fiber treated with 5% NaOH and 2% $KMnO_4$, which resulted in the microfiber size of the water chestnut fiber being 3.84 - 4.99 μm and 2.858 - 7.346 μm, while fiber size without treatment 3.247 - 10.66 μm (Ninis H. Haryanti *et al.* 2021).



(a) (b) (c)

Fig. 1. (a) Fiber Size with 6% NaOH treatment, (b) Bleaching before ultra-sonication, (c) After ultra-sonication.

The 6% NaOH treatment had an effect on the chemical components of the water chestnut fiber, as well as the ultra-sonication process. This can be seen through the FTIR spectra, as shown in Fig. 2. This spectrum determines the functional groups of the chemical components in the water chestnut fiber by treating with 6% NaOH alkalization, bleaching before ultra-sonication and after ultra-sonication.

Cellulose is present at the peak of the wave number 3334 cm^{-1} in the detected O-H group. The intensity of this group is influenced by the treatment given to the fiber (Nomanbhay *et al.*, 2013). The absorption intensity of water chesnut fiber after bleaching into microfiber before and after the ultra-sonication process was greater than in the 6% NaOH treatment before ultra-sonication.

From the FTIR graph, it can be seen that the absorption peak on the O-H bond for fibers treated with bleaching into microfiber of water chestnut before ultra-sonication has a gentler wave than the 6% NaOH treatment, because the fiber contains a lot of water in the O-H group.

After the ultra-sonication process, heating occurs so that the water is reduced, meaning that the ultra-sonication process has succeeded in reducing the percentage of excess water content before ultra-sonication. Cellulose is also present at wave number 2914 cm^{-1} has C-H groups (Rachmawaty *et al.*, 2013) and wave number is 1636 cm^{-1}, 1371 cm^{-1}, 1312 cm^{-1} and the absorption area of -glycosidic cellulose bonds is shown at number wave 895 cm-1 (Nomanbhay *et al.*, 2013).

The lignin content can be seen in the FTIR analysis in Fig. 2 with a wave of 1636 cm^{-1} and 1633 cm^{-1} the C-H group can also indicate the presence of lignin absorbed by water (Maslahat *et al.*, 2016), then at a wave of 1031 cm^{-1} the presence of a lignin group. C-O indicates lignin (Nomanbhay *et al.*, 2013),

This aromatic ring spectral band is also called amorphous absorption band. In the 664 cm^{-1} wave which shows the aromatic C-H functional group of lignin (Fatriasari & Hermiati, 2016) and the hemicellulose content in the 1371 cm^{-1} wave (Nomanbhay *et al.*, 2013).

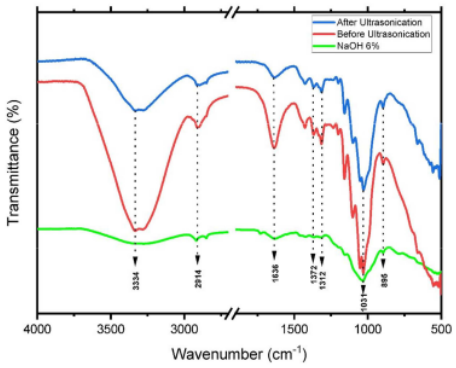


Fig. 2. FTIR spectra of 6% NaOH alkalized water chestnut fiber and microfibers before and after ultrasonication.

The difference in the intensity of each functional group from the IR graph caused differences in the chemical, physical and mechanical properties of the water chestnut fibers from the three treatments. Chemical, physical and mechanical properties of untreated water chestnut long fiber, treated with 6% NaOH, water chestnut microfiber by bleaching before and after ultra-sonication are presented in Table 1 and Table 2. Cellulose is the main structural component of cell walls of green plants and is an organic compound. Based on Table 1, the cellulose content of long fiber water chestnut without and with treatment had an average cellulose content of 54.74% and decreased to 50.94% after NaOH treatment.

Table 1. Chemical content of water chestnut fiber before and after 6% NaOH alkalization.

Alkalizing by 6% NaOH	Sample	Percentage (%)		
		Lignin	Cellulose	Hemicellulose
Before	1	54.98	54.54	1.67
	2	42.18	54.29	3.36
	3	37.93	55.40	2.47
	Average	45.03	54.74	2.50
After	1	9.03	53.10	0.33
	2	9.42	43.53	0.36
	3	9.92	56.19	0.31
	Average	9.45	50.94	0.33

Table 2. The results of moisture content, density, and tensile strength of long fiber water chestnut before and after ultra-sonication.

	Sample	Moisture content (%)	Density (g/cm3)	Tensile strength (MPa)
Without treatment	1	15.09	0.09	264.36
	2	14.40	0.06	247.70
	3	14.93	0.08	550.77
	Average	14.81	0.08	354.28
6% NaOH treatment	1	7.83	1.20	3,538.57
	2	7.36	1.28	3,249.71
	3	7.66	1.35	3,058.02
	Average	7.62	1.28	3,282.10
Microfiber before Ultra sonication	1	5.07	0.48	-
	2	5.48	0.47	-
	3	4.82	0.46	-
	Average	5.12	0.47	-
Microfiber after Ultra sonication	1	4.28	0.19	-
	2	4.10	0.22	-
	3	4.59	0.19	-
	Average	4.32	0.20	-

The yield of cellulose content obtained is greater than that of alkalization using 2% KMnO4 and 5% NaOH, which is 22.58% and 19.10%, respectively (Ninis H. Haryanti *et al.*, 2021). The decrease in cellulose content is caused by degradation through alkali or what is called "peeling off" (Lismeri *et al.*, 2016). The decrease in cellulose content can also be caused by the concentration of alkali and the duration of immersion, causing damage to the cellulose element (Jenifer *et al.*, 2020).

In water chestnut fiber, the cellulose content should remain stable or not decrease too much in order to give strength to the fiber itself and also not be easily degraded chemically or mechanically.

Analysis of water chestnut lignin levels in without treatment obtained an average of 45.03%, while with 6% NaOH treatment there was a significant decrease of 9.45% (see Table 1). This is due to the faster lignin bond breaking reaction. Lignin is one type of organic polymer that plays an important role in plant structure; lignin fills the space from within the cell wall between cellulose, hemicellulose and pectin. Lignin is hydrophobic i.e. insoluble in water and stable in nature and acts as a "glue" that connects cellulose and hemicellulose. In the utilization of natural fiber as a composite reinforcement material, excess lignin content can affect the quality of the composite. The presence of an amorphous structure of lignin in water chestnut plants can be destroyed and damaged by using an alkaline solution. This is in accordance with the purpose of the alkaline treatment, which is to reduce the lignin content of the water chestnut plant so that it still provides stiffness and protection for the fiber. Excess lignin content can also affect the use of natural fibers as a composite mixture. Changes in the lignin content when used as a composite material will improve the interface between natural fibers as reinforcement and the matrix to be stronger, because lignin causes the fiber-matrix adhesion force to become weak (Syarief, 2011; Ninis H. Haryanti *et al.*, 2021).

Analysis of hemicellulose content obtained an average of 2.50% without treatment, while with 6% alkaline NaOH treatment the average was 0.33%. These results are in accordance with the objective of isolating cellulose from lignin and hemicellulose. Hemicellulose is part of the composition of the water chestnut plant which acts as a natural matrix and is one of the constituents of plant cell walls in addition to cellulose and lignin. Hemicellulose is needed to strengthen cell walls through interactions with cellulose and lignin. Cellulose extraction will reduce hemicellulose content in long fiber of water chestnut. Hemicellulose content decreased with treatment and increased NaOH concentration. With the alkalizing treatment and the more concentration of the treatment in the solution, the bonds that connect hemicellulose with cellulose and lignin are broken, namely hydrogen bonds (Winarsih, 2016; Haryanti *et al.*, 2021).

The water chestnut plant has a high-water content because it grows in swamps. High moisture content in the fiber can reduce the quality of the composite. Harsono, (2013) explained that freshly removed water chestnut has a high water content, which is around 91.7432%, so that its use as a composite reinforcement must be reduced to less than 15% according to SNI 06-3730-1995 standards.

The decrease in lignin and hemicellulose content was followed by a decrease in the water content of the water chestnut fiber, as shown in Table 2. The average water content of the water chestnut long fiber without treatment was 14.81% and after 6% NaOH alkalization treatment it became 7.62%. The water content with this treatment showed much lower results than the alkalization with 5% NaOH and 2% KMnO4 which was 18.31% and 17.11%, respectively (Haryanti *et al.*, 2021). The reduction in water content after alkaline treatment with NaOH is due to NaOH reacting with the fiber and releasing hydrogen bonds in the fiber structure network (Boimau & Cunha, 2015).

The water content of water chestnut microfiber with bleaching treatment resulted in a reduction in water content from before ultra-sonication, at an average of 5.12% and after ultra-sonication at an average of 4.32% as shown in Table 2. The treatments that have been carried out have met the SNI standard. 06-3730-1995 and is suitable for use as a composite reinforcement material because the value is below 10% (Sahara *et al.*, 2017; Hairiyah *et al.*, 2017).

Natural fibers have a lower density when compared to synthetic fibers, so natural fibers can only be used as lightweight composite reinforcement materials. The physical density of the fiber is indicated by its density value. Without treatment, the average density of water chestnut fiber was 0.08 g/cm^3 and increased after 6% NaOH treatment was 1.28 g/cm^3.

Water chestnut fiber in a dry state will float if placed in water and the absorption is not perfect to water and this shows that its density is low. Meanwhile, after the NaOH treatment, there was a binding of OH compounds on the water chestnut fiber and OH on NaOH so that the specific gravity of the water chestnut fiber increased (Hashim *et al*., 2017).

Likewise, the mean density of water chestnut microfiber before ultra-sonication increased to 0.47 g/cm^3 and after ultra-sonication the average density became 0.20 g/cm^3. The smaller the value of the density of the water chestnut fiber, the lighter the composite will be obtained. The decrease in sample density was caused by the smaller fiber size and the cleaner fibers due to the ultra-sonication process. The decrease in density by the bleaching and ultra-sonication processes occurred because these processes were effective in reducing lignin and/or hemicellulose in water chestnut microfibers. The removal of lignin and hemicellulose was carried out gradually, thereby increasing the decrease in density. The bleaching process breaks the hydrogen bonds between the hydroxyl groups (-OH) of cellulose, lignin, and hemicellulose, causing defibrillation so that the fibers become smaller as shown in Fig. 2 (Roy *et al*., 2012).

Mechanical properties can be defined as the response or behavior of a material to a certain load, and can be in the form of force, torque, or a combination of both. The mechanical properties of fiber can be expressed in several parameters, including tensile strength. Natural fibers can be treated in various ways to produce reinforcing materials with different mechanical properties.

At small to nano fiber sizes, crystalline cellulose can be developed in the form of composite materials. In general, the smaller the fiber diameter, the higher the composite strength. Besides that, the shape of the fiber used for the manufacture of composites does not really affect it, what affects is the diameter of the fiber, in addition to the shape the fiber content also affects the length and diameter of the fiber which greatly affects the tensile strength and strain strength. The length of the fiber to the diameter of the fiber is known as the aspect ratio. The greater the aspect ratio, the greater the tensile strength of the fiber in the composite (Schwartz, 1984). So the longer and the greater the strain.

The results of the analysis of the tensile strength of water chestnut long fiber are shown in Table 2. Without treatment, the average tensile strength of water chestnut fiber was 354.28 Mpa and a significant increase occurred after 6% NaOH alkaline treatment, which was 3,282.10 MPa. This difference in tensile strength concluded that the alkalization treatment was able to increase the tensile strength of the long fiber of water chestnut so that the purpose of this treatment was achieved by increasing the tensile strength of the fiber.

Hydrophilic properties are a major problem for natural fibers when used as reinforcement in composite materials. The moisture content of the fiber, depending on the non-crystalline part and the content of voids or voids. The hydrophilic nature of natural fibers affects the overall mechanical properties as well as the physical properties of the fiber.

This hydrophilic nature must be considered when using long fibers of water chestnut. It is hoped that water chestnut long fiber can be used in the manufacture of composites and provide added value economically. Besides that, the length of the fiber is one of the factors that affect the performance or strength of the composite where the longer the fiber in the matrix, the surface of the fiber that bears the load given by the matrix becomes larger and vice versa.

Conclusion

This study showed that the alkalizing treatment of 6% NaOH had decreased the levels of lignin, cellulose, and hemicellulose as well as the moisture content of water chestnut fiber. This treatment has increased the density and tensile strength of water chestnut fibers. The bleaching and ultra-sonication processes also increase the density of the microfiber of water chestnut. The diameter of the water chestnut microfiber after the bleaching and ultra-sonication process was smaller than before the ultra-sonication.

The size of the fiber after 6% NaOH treatment was 373.05 μm while the fiber after becoming microfiber was 4.28-5.96 μm before ultra-sonication and 3.17-4.00 μm after ultra-sonication. Treatment of 6% NaOH and bleaching and ultra-sonication were better than before treatment. Likewise with the manufacture of water chestnut in the form of microfiber, it is hoped that this water chestnut microfiber when used as a reinforcing material in the manufacture of composites will blend better.

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