MODIFICATION OF PURUN TIKUS (ELEOCHARIS DULCIS) LONG FIBER WITH POTENTIAL AS COMPOSITE REINFORCEMENT MATERIAL

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Modification of Purun Tikus (*Eleocharis dulcis*) Long Fiber with Potential as Composite Reinforcement Material

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Abstract. The choice of natural fiber purun tikus (*Eleocharis dulcis*) as a composite reinforcement material is related to its benefits, such as low price, abundant quantity, renewable, and environmentally friendly. In this research, the effect of alkalizing treatment and hot water immersion on the properties of purun tikus fiber were investigated. The long fiber of purun tikus measuring 20 cm and diameter $\leq$ 0.064 cm was modified with 120°C hot water immersion and 5% NaOH immersion for 1 hour each. These treatments were applied to improve the properties of purun tikus fiber, such as their properties of chemical (moisture, lignin, cellulose, and hemicellulose), physical (density), and mechanical (tensile strength). The results showed a decrease in the water content, lignin, cellulose, and hemicellulose of purun tikus fiber, while the density and tensile strength increased after the treatment of 120°C hot water immersion and NaOH 5% alkalization. The NaOH 5% alkaline treatment was better than 120°C hot water immersion in terms of reducing water content, lignin, cellulose, and hemicellulose. Although the tensile strength of the modified NaOH 5% NaOH purun tikus fibers is lower than the 120°C hot water immersion treatment, it was still higher than before the modification. It is shown that these fibers blend well in the manufacture of composite materials and improve their mechanical properties.

1. Introduction

Composites are made from a combination of two or more materials which were macroscopically different. Composites have advantages compared to other materials, which are stronger, stiffer, lighter, corrosion resistant and economical [1]. Reinforcing materials for composites usually use synthetic fibers, but their use causes many problems and the price is quite expensive, so the manufacturing industry is starting to search for a substitute. Many studies have been carried out using natural fibers as a reinforcing material in composite because natural fibers have good mechanical properties, low density, are easy to recycle, and are biodegradable [2]. Purun Tikus could be used as a candidate for that natural fiber reinforcing.

Purun tikus (*Eleocharis dulcis*) is a weed that grows in swampy areas that can be used as a constituent of composite materials because it has a fairly high natural fiber content [3]. The province of South Kalimantan has a fairly large availability of natural fiber purun tikus. In the right condition, the purun tikus can grow quickly. The stem height is about 90-150 cm, with an erect tubular stem. Sunardi [4] stated that purun tikus has a cellulose content of around 40%, which can be extracted and used for various useful materials [4]. The use of purun tikus fiber as a composite material provides an extra advantage because it can increase the economic value of the fiber. Several studies on purun tikus and their use have been carried out, such as handicrafts, food for swamp buffalo, biofilters, heavy metal absorbers and cement board composite materials [5-9].

Before purun tikus can be used as a reinforcing material, it must be processed or modified first to reduce unnecessary fiber elements. The alkali treatment method is the most commonly used method because it has been proven to improve the mechanical properties of the fiber, and is also effective in removing layers of pectin, lignin, hemicellulose and other impurities present in the fiber [10]. Oroh [11] conducted research on the analysis of the mechanical properties of composite materials from coconut fiber using a 5% NaOH treatment, which showed that there was an increase in the bending strength of the composite. From several studies on the modification of the fiber, no one has used 120°C hot water immersion and 5% NaOH with 1 hour immersion time. Besides that, modification research using purun tikus fiber, 20 cm in length and less than 0.064 cm in diameter, as a composite material has not been carried out.

The size of the natural fiber to be used is important in the manufacture of composite materials. The length and diameter of the fiber affect the tensile strength and strain [12,13]. Longer fibers and smaller diameters will have a larger surface fiber that will carry the load, so the composite would be stronger. This is related to the aspect ratio, which is the ratio of fiber length to fiber diameter. Mahmuda [12] conducted a study on three variations of fiber length and found that the largest stress and strain values were in the longest fiber variation.

In this research, the long fiber of purun tikus was modified by providing hot water immersion and alkaline treatment to the fiber to improve its mechanical properties. The chemical, physical, and mechanical properties of the long fiber of purun tikus produced from the modification between 120°C hot water immersion and 5% NaOH with an immersion time of one hour will be studied.

2. Methods

2.1 Preparation of Sample

This research is experimental research conducted in a laboratory. Purun tikus that have a length in the range of 100-160 cm are dried for two days and then cut into pieces with a size of 20 cm. After that, it was split in half and combed to get fine fibers with a diameter of less than 0.064 cm. Purun tikus fibers were then separated into two treatments. First, soaking in hot water at 120°C for 1 hour and, secondly, soaking in 5% NaOH solution for one hour. Furthermore, the samples were wind-dried at room temperature and ready to be measured for their chemical, physical, and mechanical properties.

2.2 Experimental Methods

The water content in purun tikus fiber will determine its quality as a natural fiber of composite material. Referring to SNI 06-3730-1995, the maximum water content must be less than 15% [14]. The percentage of water content in purun tikus was calculated by equation (1):

$$KA\ (\%) = \frac{A-B}{C}\, x\ 100\% \qquad (1)$$

where KA is moisture content (%), A = mass (sample + cup) before drying; B = mass (sample + cup) after drying and C = mass of sample

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

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

where A = weight of lignin precipitate; B = dry sample weight

Cellulose content was calculated as a percentage of cellulose in purun tikus fiber, based on SNI 14-0444-1989, referring to equation (3):

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

where A = weight of cellulose precipitate; B = dry sample weight

The hemicellulose content was calculated according to SNI 14-0444-1989, referring to equation (4)

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

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).

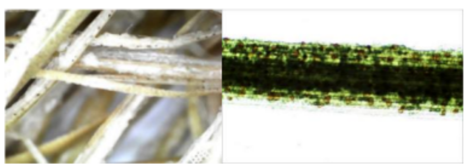
Purun tikus density was calculated by the ratio of the mass and volume of the increase in water using an equation (5). The mass of purun tikus sample was weighed and then immersed in water. The increase in water volume was measured. The density was calculated referring to equation (5):

$$\rho = \frac{M}{V} \tag{5}$$

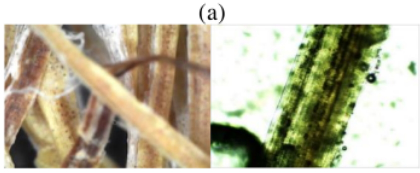
where M = fiber weight (grams); V = Volume added of water (ml)

3. Results and Discussion

The measurement of the long fiber diameter of purun tikus was carried out with a microscope, and the results are shown in Figures 1(a) and (b). Photos of the measurement results of purun tikus fiber size from 2 types of sample treatments, namely Figure 1(a), 120°C hot water immersion and Figure 1(b), 5% NaOH treatment.



(a)



(b)

Figure 1. Photo of purun tikus fiber after treatment (a) 120°C hot water immersion and (b) 5% NaOH treatment

Purun tikus fiber with hot water immersion treatment has a size of 311.51 μm and also there are still a lot of dirt on the fiber. While for the 5% NaOH treatment, the fiber size is 324.34 μm, and it looks like only a little dirt remains. This concluded that the alkaline treatment was able to remove impurities in the fiber. Purun tikus fiber with hot water immersion has a smaller diameter than the 5% NaOH treatment. This is indicated that the OH content in cellulose due to hot water immersion is reduced. It evaporates during treatment in the form of water vapour, while in NaOH treatment, OH compounds increase due to the absorption of OH from NaOH. This is also shown by the FTIR results in Figure 2, which is at a wave number of 3301 cm^{-1}, where the absorption intensity in the NaOH treatment is greater than in hot water immersion. Overall functional groups of purun tikus fiber for these two treatments are shown in Figure 2 and Table 1.

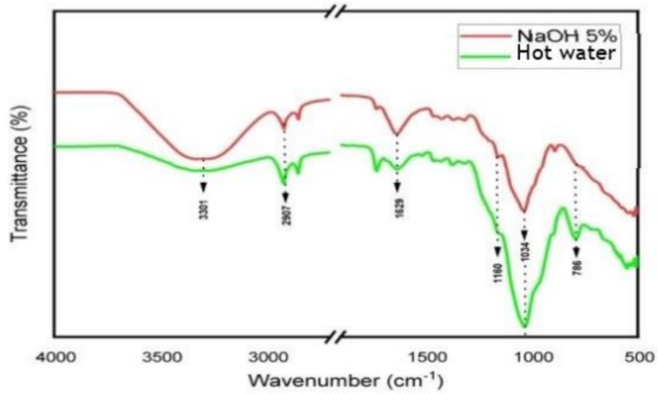


Figure 2. FTIR of purun tikus fiber in 120°C hot water immersion and 5% NaOH treatment

Table 1. IR wavenumber area of purun tikus fiber in 120°C hot water immersion and 5% NaOH alkalization

Wavenumbers (cm^{-1})		
Hot water immersion	**5% NaOH**	**Bonding and Functional Groups**
3301	3301	O-H stretching [15]
2907	2907	C-H stretching [15]
2848	2848	C–H symmetrical stretching [16]
1725	1725	C=O stretching vibration [16]
1629	1629	O-H bending of absorbed water [16]
1160	1160	C-O-C asymmetrical stretching [16]
1034	1033	Aromatic C-H in-plane deformation, C-O deformation; primary alcohol [16]
786	-	C-H deformation [17]

The peak wavenumber between 3000 and 3500 cm^{-1} is the strain vibration of the OH group of cellulose. The intensity of this group is influenced by the treatment given to the fiber. In alkaline treatment, assisted by microwaves, the intensity is reduced [18]. From the FTIR graph, it can be seen that at the absorption peak in the O-H bond, the fiber with 5% NaOH treatment has a gentler wave than the hot water immersion. This is because the 5% NaOH immersion absorbs more water. The effect of this treatment is also shown by the absorption peak of 1629 cm^{-1}, which is related to the O-H bending vibration during water absorption [16]. The peak wavenumber 2907 cm^{-1} is related to the C-H strain vibration that occurs in the methyl and methylene groups [18]. The peak of the wavenumber 2848 cm^{-1} has indicated that the bonding is C-H symmetrical stretching. In this bond, there is a symmetrical stretching that occurs in the C-H bond [16]. From the graphs obtained at the two absorption peaks of 2907 cm^{-1} and 2848 cm^{-1}, it can be said that the presence of cellulose is still detected. The peak of the 1725 cm^{-1} wavenumber corresponds to the C=O strain bond, which indicates the presence of hemicellulose at the 1725 cm^{-1} wave [16]. In the hot water immersion, the peak of this wave is more gentle. This indicates that the hemicellulose content is higher than the 5% NaOH treatment, meaning that the 5% NaOH treatment reduces the hemicellulose content in the fiber. The peak wavenumber 1160 cm^{-1} is associated with the stretching of the C-O-C bond [16], which is related to the structure of the lignin that makes up wood [19]. The peak wavenumber 1033-1034 cm^{-1} is associated with aromatic

C-H vibrations on plane deformation and C-O deformation [16]. At the peak of the wavenumber 786 cm^{-1} associated with the C-H bond deformation, the absorption peak in this wave only appears in the fiber by immersion in hot water, which indicates that there is a deformation of cellulose and hemicellulose in the polysaccharide bonds.

The difference in the intensity of each functional group on the IR graph caused differences in the chemical, physical and mechanical properties of purun tikus fibers of the two treatments. Table 2 shows the chemical, physical and mechanical properties of purun tikus fiber, for the untreated, hot water treatment and 5% NaOH immersion.

Table 2. Moisture content, lignin, cellulose, hemicellulose, density, and tensile strength of purun tikus fiber (without treatment, 120°C hot water and 5% NaOH immersion)

Treatment	Sample	Content (%)				Density (g/cm^3)	Tensile strength (MPa)
		Water	Lignin	Cellulose	Hemi-cellulose		
Without treatment	1	15.09	54.98	54.54	1.67	0.32	4.14
	2	14.40	42.19	54.29	3.36	0.30	4.24
	3	14.93	37.93	55.39	2.47	0.26	4.80
	Avg	**14.77**	**45.03**	**54.74**	**2.50**	**0.29**	**4.39**
Hot Water	1	9.90	33.88	43.58	2.18	0.59	100.11
	2	9.50	31.97	40.28	2.38	0.71	47.27
	3	10.20	35.24	49.16	2.46	0.66	108.28
	Avg	**9.87**	**33.69**	**44.34**	**2.34**	**0.65**	**85.22**
5% NaOH	1	9.02	19.79	42.39	0.25	1.39	24.06
	2	8.70	21.12	43.60	0.40	1.50	38.01
	3	8.91	18.62	42.50	0.33	1.33	37.89
	Avg	**8.88**	**19.84**	**42.82**	**0.33**	**1.40**	**33.32**

Analysis of lignin content showed that the hot water immersion treatment and 5% NaOH treatment had reduced the lignin content of the purun tikus fiber. Haryanti [3] stated that the lignin content in the purun tikus short fiber without treatment was 45.03%. Based on Table 2, both treatments succeeded in reducing the lignin content in the fiber, but the NaOH treatment was the best treatment in reducing the lignin content in the purun tikus fiber. Reduction of lignin content is very necessary if the fiber is to be used as a composite reinforcement so that the fiber can easily bind to other materials in the manufacture of composite materials. Boopathi [20] also used NaOH treatment to reduce lignin levels. NaOH compounds are usually used to remove dirt or lignin from fibers. This alkaline treatment affects the surface of natural fibers, namely cellulose, which has an optimum water content which can be reduced to obtain natural properties [1,21]. Haryanti [3] obtained lignin content of purun tikus short fiber with 5% NaOH treatment for 3 hours at 13.96%, Sunardi & Istikowati [4] obtained purun tikus lignin content of 26.40%. The lignin content obtained by these two studies was not too large as compared to the lignin content in wood, which ranged from 23-35%. While in this study, the lignin content produced in the fiber by soaking in hot water was quite large at 33.69% and in the 5% NaOH treatment, the lignin content produced was quite small at 19.84%.

In addition to reducing lignin levels, these two treatments also reduced hemicellulose levels in purun tikus fiber. Without treatment, the lignin content was 2.50%, while by the hot water immersion, it was obtained at 2.34%, and NaOH treatment was 0.33%. Delignification is carried out to increase the cellulose content and reduce the hemicellulose and lignin content found in plants [22]. This is in accordance with the purpose of the treatment, which is to isolate cellulose from lignin and

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hemicellulose. The hemicellulose content of short fiber purun tikus with 5% NaOH treatment for three hours was 0.97% [3], while in this study, the hemicellulose content of long fiber with the 5% NaOH treatment was 0.33%. This value is smaller than in previous studies. Immersion in hot water at a temperature of 120°C has also reduced the hemicellulose content by 2.34%.

The cellulose content of purun tikus fiber without soaking was about 54.74% and decreased after being treated, which was about 42.82% for the 5% NaOH treatment and 44.34% for the hot water immersion. The decrease in cellulose content indicated that during the alkalization and soaking process, a small portion of the cellulose component was dissolved. Alkalization process has removed impurities in natural fibers, which are lignin and pectin and gave rise to cellulose, thereby increasing the mechanical properties of the fiber [15,23]. Haryanti [3] found that the cellulose content of purun tikus short fiber with the 5% NaOH treatment for three hours was 19.10%. The main advantages of using cellulose as a reinforcing material in polymer matrices are low density, not easily abraded, producing high stiffness properties, easy to recycle, the material is easy to obtain because it is widely available in nature, and cheap [15].

The average untreated purun tikus fiber density was 0.29 g/cm^3, and with the 5% NaOH treatment was 1.40 g/cm^3. The density of purun tikus before treatment was low because it was not perfect in absorbing water, so the fiber seemed to float. Alkali treatment can increase the density of the fiber, both by the concentration of NaOH and the length of the soaking process. This treatment can affect the specific gravity [24]. The results of the analysis of the fiber density of purun tikus without treatment were 0.29 g/cm^3, and with the hot water immersion was 0.65 g/cm^3, and the 5% NaOH immersion was 1.40 g/cm^3. This indicates that both the hot water immersion and the 5% NaOH treatment can increase fiber density.

Furthermore, its mechanical properties, and the tensile strength of purun tikus fiber increased after treatment. Without treatment, the fiber tensile strength was 4.39 MPa, with the hot water immersion 85.22 MPa and with the 5% NaOH treatment 33.32 MPa (Table 2). Tensile strength properties with the hot water immersion appear higher than the NaOH treatment. This is indicated because the reduction in the lignin content, which is a reinforcement in the fiber is more than by immersion. This alkalization directly affects the structure of the fiber, and provides a better bond between the fiber and the matrix, which in turn will improve the mechanical properties of the composite [25]. However, the hydrophilic nature of lignin is a major problem for natural fibers when used as reinforcement in composite materials. The hydrophilic nature of these natural fibers affects the overall mechanical properties of the fiber as well as its physical properties, and this property should be a concern when using purun tikus fibers.

4. Conclusion

Modification of long fibers of purun tikus, 120°C hot water immersion and 5% NaOH treatment have reduced water content, lignin, cellulose, and hemicellulose. The modification has increased the density and tensile strength of the fiber compared to before the treatment. Treatment of 5% NaOH immersion on long fiber of purun tikus is better than treatment of 120°C hot water immersion, based on the chemical and physical properties of purun tikus fiber such as water content, lignin, cellulose, hemicellulose and density, but on mechanical properties, tensile strength, 120°C hot water immersion was better. For 120°C hot water and 5% NaOH immersion, fiber sizes were 311.51 μm and 324.34 μm, respectively. Based on the results of these modifications, purun tikus long fiber can be used as a reinforcing material in the manufacture of composites.

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