

AIMS Materials Science, 11(6): 1199–1219. DOI: 10.3934/matersci.2024059 Received: 15 July 2024 Revised: 02 December 2024 Accepted: 09 December 2024 Published: 23 December 2024

http://www.aimspress.com/journal/Materials

Research article

Characteristics of water chestnut (Eleocharis dulcis) long fiber reinforced

composite modified by NaOH and hot water

Ninis Hadi Haryanti¹, Suryajaya^{1,*}, Tetti Novalina Manik¹, Khaipanurani¹, Adik Bahanawan² and Setiawan Khoirul Himmi³

- ¹ Program Studi Fisika FMIPA, Universitas Lambung Mangkurat, Banjarbaru, 70714, Indonesia
- ² Research Center for Biomass and Bioproduct, National Research and Innovation Agency (BRIN), Jl. Raya Bogor km. 46 Cibinong, Bogor, 14911, Indonesia
- ³ Research Organization for Life Sciences and Environment, National Research and Innovation Agency (BRIN), Jl. Raya Bogor km. 46 Cibinong, Bogor, 14911, Indonesia
- * Correspondence: Email: Suryajaya@ulm.ac.id; Tel: +62-813-5194-1407.

Abstract: The length and diameter of the reinforcing fibers in a composite greatly influence its tensile strength. This research investigated the physical and mechanical properties of composites reinforced with water chestnut (*Eleocharis dulcis*) long fibers. The fibers were modified with 5% NaOH and hot water (100 °C), each for one hour. Polyester was used as a matrix. The test refers to ASTM (American Society for Testing Materials) standards D638 and D790. The composite was made using the hand lay-up and vacuum bagging method. The volume fractions of water chestnut long fiber, e-glass, and polyester samples were 40%:0%:60%, 20%:20%:60%, 0%:40%:60%, 10%:30%:60%, and 30%:10%:60% (A, B, C, D, and E, respectively). The average density of water chestnut–reinforced composite modified by 100 °C hot water immersion and 5% NaOH treatment was 1.61–2.20 and 1.24–2.57 g/cm³, respectively. The tensile test results show that sample D had the highest average tensile strength at 134.68 and 130.13 MPa following immersion in hot water and 5% NaOH, respectively. The highest average modulus of elasticity (MoE) and modulus of rupture (MoR) for the modified composites were in sample C, at 7,580 and 235.53 MPa.

Keywords: composite; e-glass; polyester; water chestnut fiber (Eleocharis dulcis)

In general, composites are composed of a matrix and reinforcement. The matrix can be polymer, ceramic, or metal [1]. Polymer matrix, either thermosetting or thermoplastic plastic matrix, is the most widely used. Thermosets are more popular than thermoplastics because of their higher strength and resistance to high temperatures. In this study, polyester is used. Polyester can be either thermoplastic or thermoset. Polyester resin becomes hard when mixed with a hardener (thermoset) [2].

The composite reinforcements can be particles, fibers (short or continuous), and lamina. The fiber reinforcements are divided into two categories: natural and synthetic [3]. Polypropylene, polyethylene, polyester, nylon, carbon, aramid, and acrylic are often used as synthetic fiber reinforcements [4]. When these synthetic reinforcements are incorporated into a polymer matrix, fiber-reinforced polymer (FRP) composites such as carbon fiber-reinforced polymer (CFRP), aramid fiber-reinforced polymer (AFRP), and glass fiber-reinforced polymer (GFRP) are formed [5]. Synthetic reinforcements have the advantages of being cheap, lightweight, mechanically strong, and corrosion-resistant but are less environmentally friendly [5]. Therefore, in recent years, various researchers have shown interest in using natural fibers as composite reinforcements [6]. This is because natural fibers have the potential to be available in nature, have low density, can be recycled, are cheap, and have good mechanical properties [3]. Plants such as cotton, flax, jute, hemp, sisal, pineapple, kenaf, ramie, banana, or bamboo and wood are used as reinforcements of composite structures [4,6]. Those natural fibers are much cheaper than synthetic fibers like glass, nylon, or carbon. The low density of natural fibers allows the fabrication of composites that combine good mechanical properties with low specific mass. The mechanical properties will be varied depending on the fibers used. However, natural fiber composites have much lower mechanical properties than their synthetic counterparts and are not fireproof.

South Kalimantan has many sources of natural fiber, such as water chestnut, water hyacinth, bundung, bemban, and others. Usually, water chestnut (*Eleocharis dulcis*) is found growing in tidal swamps, lowland swamps, and even rice fields. Water chestnut grows well in acidic soils with a pH of 2.5–3.5 [7]. This plant has quite strong fibers because of its rope-like fiber texture that can exceed rattan or bamboo tensile strength [8,9]. Local people exclusively use chestnuts to make mats and bags. In previous research, water chestnuts have been explored for use as a biofilter [10], activated carbon [11], and cement board composite material [12].

One of the main disadvantages of using natural fibers as composite reinforcements is their hydrophilic nature, which causes natural fibers to absorb high levels of moisture [13]. This makes them inconsistent with the hydrophobic matrix and leads to weak interfacial bonding between the fibers and polymers. Surface modification using various chemical compounds is the alternative way to improve their mechanical and physical properties. Before being used as a natural fiber reinforcement, water chestnut must be processed or modified to blend with the matrix by reducing unused fiber elements. Alkaline treatment is the most commonly used method for modifying natural fibers because it can improve the adhesion properties or interfacial attachment of natural fibers with the composite matrix, which in turn can increase the tensile strength of the composite. In previous research, 5% NaOH base treatment was used to alkalize water chestnut and reduce the lignin, hemicellulose, and cellulose content of its fibers, which in turn reduced fiber size [14–18]. The length of soaking in NaOH can also affect the water content of the composite. As the soaking time increases, the water content in the composite also increases [19].

The size of the natural fiber to be used is essential in making composite materials. The fiber's length and diameter greatly influence the tensile strength and strain of the composite [20]. The smaller the diameter of the fiber, will increase its tensile strength. This occurs because the voids in the fiber are small and there are more bonds between molecules, so the strength is greater [21]. The longer the fiber and the smaller the diameter, the stronger the composite will be because the fiber's surface that can carry the load will also be more significant. This is also related to the ratio of fiber length and diameter.

In addition, the combination of natural and synthetic fibers in the same matrix (hybrid composites) is also expected to improve its mechanical properties or other properties [22,23]. This is a potential advantage of the combined inherent attributes of each fiber. Therefore, apart from being modified, the water chestnut fiber will be combined with a synthetic fiber, e-glass. E-glass and polyester resin are often used as composite materials. E-glass is a type of glass fiber with a low alkali glass concentration, while polyester resin is cheaper and can bond with natural fibers without causing reactions and gases [24–26]. In previous research, hybrid composites, such as hybrid composites of bamboo and glass fibers [23] or banana/glass fiber-reinforced polypropylene hybrids [22], have been shown to improve the mechanical properties of composites and be resistant to the environment.

Processing with hot water is inspired by the procedure for making mats carried out by local people. Water chestnut is first boiled and dried before being woven into mats. When it is heated, it becomes soft. Many studies have shown that when heated, natural fibers are thermally degraded through dehydration, depolymerization, and oxidation. At around 100 °C, fibers' components lignin and hemicellulose will be damaged due to the breaking of their chemical chains [27,28]. Therefore, the cellulose structure is destabilized, and fibers become soft. Several works (e.g., [13,29,30]) show that soaking in hot water can change the tensile strength of natural fibers. This is a consideration when making a hybrid composite of long fiber water chestnut with synthetic fiber e-glass and polyester to obtain a composite with good mechanical properties. So far, no composite studies have used long water chestnut fibers modified with 5% NaOH and soaked in hot water (100 °C) for 1 h each, and then hybridized with e-glass fibers. In addition, research on fiber-reinforced composites with a water chestnut length of 20 cm and a diameter of ≤ 0.064 cm has never been carried out.

The characteristics of hybrid composites depend on the composition of the mixture of matrix and reinforcement. The use of long water chestnut fibers as reinforcing material could determine the properties of the composite, which transmits the load distributed by the matrix. It is hypothesized that long water chestnut fibers could produce physical and mechanical properties of hybrid composites that meet the Indonesian National Standard (SNI). This research was carried out to obtain the physical and mechanical properties of a hybrid composite reinforced with long water chestnut fibers and e-glass with a polyester matrix. Long water chestnut fibers were produced by a modified treatment of soaking in 5% NaOH solution and hot water with a soaking time of 1 h each.

2. Materials and methods

2.1. Preparation of water chestnut long fibers

The water chestnut (*Eleocaris dulcis*) used was obtained from the area around the Lambung Mangkurat University campus in Banjarmasin. Water chestnuts with a length of 100–160 cm were cleaned and dried in the sun twice for 8 h. To obtain long fibers, dried water chestnuts were cut into 20 cm long sections, split into two parts, and then combed until smooth. The long fibers obtained were 19 cm

long and 0.064 cm in diameter. Before being ready to be used as a reinforcing material, long water chestnut fibers need to be modified. This modification was performed by soaking in a 5% NaOH solution or hot water at 100 °C, each for 1 h.

2.2. Hybrid composite compositions

The matrix used in this research was Yukalac 157 BTQN-EX polyester resin with a catalyst (hardener) MEKPO (methyl ethyl ketone peroxide) in a 2:1 ratio. The polyester matrix had a density of 1.217 g/cm³ with a tensile strength of 24.4 MPa [31]. The synthetic fiber used was CSM e-glass fiber. Its tensile strength, compressive strength, elasticity modulus, and density were 3,445, 1,080, 73,000 MPa, and 2.58 g/cm³, respectively [32].

Hybrid composite compositions were made with variations in volume fraction composition, according to [33]. The volume fraction is calculated using Eq 1.

$$V_f = \frac{\frac{m_f}{\rho_f}}{\frac{m_f}{\rho_f} - \frac{m_m}{\rho_m}} \tag{1}$$

where V_f is the volume fraction of fiber, m_f is the mass of fiber, ρ_f is the density of fiber, m_m is the mass of matrix, and ρ_m is the density of matrix [33].

The variations in volume fraction composition of the long water chestnut fiber, e-glass fiber, and polyester were 40%:0%:60%, 20%:20%:60%, 0%:40%:60%, 10%:30%:60%, and 30%:10%:60%. The hybrid composite samples can be seen in Table 1.

Sample	Volume fraction (%)					
	Water chestnut long fiber	Fiber e-glass	Polyester			
A	40	0	60			
В	20	20	60			
С	0	40	60			
D	10	30	60			
Ε	30	10	60			

Table 1. Composites with variations in volume fraction composition.

The composite was made using the hand lay-up method and then vacuumed at a pressure of 4 MPa for 1 h. The mold size used was $16.5 \times 2 \times 0.5$ cm for tensile testing according to ASTM D638 and $12 \times 2.5 \times 0.5$ cm for density, modulus of elasticity (MoE), and modulus of rupture (MoR) testing according to ASTM D790. The processing process from raw materials to hybrid composites can be seen in Figure 1 below.



Figure 1. Schematic of the hybrid composite.

2.3. Physical and mechanical properties' characterization

Moisture content (%) was calculated using Eq 2. Based on the SNI 01-4449-2006 standard, the water content maximum of fiberboards is 13%.

Water content (%) =
$$\frac{initial weight-oven dry weight}{oven dry weight} \times 100\%$$
 (2)

The density of samples was calculated using Eq 3:

$$o = \frac{m}{V} \tag{3}$$

where ρ , *m*, and *V* are the density (g/cm³), mass, and volume of the sample, respectively.

1

Thickness development (%) or thickness swelling was calculated using Eq 4:

Thickness development (%) =
$$\frac{V_w - V_i}{V_i} \times 100\%$$
 (4)

where V_w is the volume of samples after immersion in the water (cm³), and V_i is the oven-dry volume of samples (cm³). Based on the SNI 01-4449-2006 standard, the thickness swelling maximum of fiberboards is 10%.

The hybrid composite materials were subjected to mechanical tests, i.e., flexural and tensile tests, using Shimadzu Autograph AGS X Universal Testing Machine 10 kN (Shimadzu Corp., Kyoto, Japan) at the Integrated Laboratory for Bioproducts, National Research and Innovation Agency (BRIN), Bogor, Indonesia. The flexural test was measured according to ASTM D790-00, which has a three-point bending system. The test was conducted to measure the ability of hybrid composite materials to withstand bending forces applied perpendicular to their longitudinal axis, called flexural strength (Figure 2). The

test was conducted at 10 mm/min and the samples were pressured until a fracture occurred. Figure 2 shows a composite specimen for the flexural test.



Figure 2. Flexural test composite according to ASTM D 790-00 [34].

The tensile test was measured according to ASTM D 638 Type I (Figure 3). Tensile properties were measured at room temperature at 2 mm/min. The results of flexural and tensile tests were analyzed to calculate the MoE, MoR, stress, and strain of the samples.



Figure 3. Tensile test composite according to ASTM D 638-14 [35].

3. Results and discussion

3.1. Characteristics of water chestnut long fiber

The chemical, physical, and mechanical properties of water chestnut fiber with and without modification, either by immersion in hot water or with NaOH solution, are presented in Table 2. Modification of water chestnut long fibers by immersion in hot water or 5% NaOH reduced water, lignin, cellulose, and hemicellulose contents [36]. This is in accordance with various studies [29,30,37–39] showing that hot water and alkalization treatments break the bonds of lignin and hemicellulose, reducing both components. As seen in Table 2, the lignin content in hot water treatment is greater than in the alkalization treatment. This means that in the hot water treatment process, less lignin is released compared to the alkalization process. It seems that the hot water treatment removes the wax layer and some lignin and hemicellulose. The remaining lignin and hemicellulose, which are the cellulose structure strengtheners, actually increase the tensile strength of the fiber. This is in line with Mamungkas' research [30], which showed that the higher the temperature, the greater the tensile strength. In the process of making mats by local people, after the water chestnut is boiled, the fibers become more elastic and can be woven into mats. Further research is needed to better understand the mechanisms that occur with different temperature treatments on the mechanical properties of fibers.

When the water, lignin, cellulose, and hemicellulose contents are reduced, the fibers' diameter decreases. Therefore, this modification increases the density and tensile strength of the fiber. Based on the results of these modifications, water chestnut's long fibers can be used as reinforcing material in making composites. The modification could also improve the compatibility between natural fibers and the matrix.

Table 2. Differences in water, lignin, cellulose, and hemicellulose content, density, and tensile strength of water chestnut without treatment, hot water immersion, and 5% NaOH treatment.

Treatment	Sample	Content (%)			Density	Tensile	
		Water	Lignin	Cellulose	Hemicellulose	(g/cm^3)	strength
							(MPa)
Without	1	15.09	54.98	54.54	1.67	0.32	4.14
treatment	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.	14.81 ± 0.27	45.03 ± 6.63	54.74 ± 0.43	2.50 ± 0.57	0.29 ± 0.02	4.39 ± 0.27
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 ± 0.24	33.69 ± 1.15	44.34 ± 3.21	2.34 ± 0.11	0.65 ± 0.04	85.22 ± 25.3
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 ± 0.12	19.84 ± 0.85	42.83 ± 0.51	0.33 ± 0.05	1.41 ± 0.06	33.32 ± 6.17

3.2. Composite characteristics

3.2.1. Composite water content

Based on the SNI 06-3730-1995 standard, the maximum water content of composites is 15%. The results of the composite water content test are shown in Table 3. The highest water content of composites occurred in sample A (40% long fiber water chestnut), and the lowest water content was obtained for sample C (40% e-glass fiber). This shows that the higher the volume fraction of water chestnut's long fibers in the composite, the higher the water content; accordingly, the higher the volume fraction of e-glass fibers, the lower the water content value. The high water content in composites with a high volume fraction of long water chestnut fibers is due to their absorption of water during the soaking process. The high water content is associated with the composite's low tensile strength, MoE, and MoR.

Treatment	Sample	Water content	Density	Thickness	Tensile strength	MoE	MoR
		(%)	(g/cm^3)	development (%)	(MPa)	(GPa)	(MPa)
Immersed in	А	1.71	1.69	2.27	25.09	0.71	14.00
hot water		1.23	1.57	2.13	23.24	0.77	18.42
(100 °C)		1.19	1.58	2.04	17.62	0.60	13.47
	Avg.	1.38 ± 0.22	1.61 ± 0.05	2.15 ± 0.08	21.98 ± 2.91	0.69 ± 0.06	15.30 ± 2.08
	В	0.68	1.89	1.92	76.05	4.13	143.48
		0.68	1.85	2.00	79.57	6.35	180.84
		0.82	2.00	1.96	126.40	3.56	36.47
	Avg.	0.72 ± 0.06	1.92 ± 0.06	1.96 ± 0.03	94.01 ± 21.59	4.68 ± 1.11	120.26 ± 55.86
	С	0.47	2.42	0.00	121.41	10.64	321.01
		0.40	2.24	0.00	100.61	6.99	282.51
		0.63	1.93	0.00	148.64	5.11	103.06
	Avg.	0.48 ± 0.08	2.20 ± 0.18	0.00	123.55 ± 16.72	7.58 ± 2.04	235.53 ± 88.31
	D	0.47	2.02	3.86	172.89	3.86	47.93
		0.51	2.00	2.00	118.52	3.04	98.30
		0.66	1.73	1.96	112.62	5.84	223.30
	Avg.	0.55 ± 0.08	2.01 ± 0.09	2.60 ± 0.83	134.68 ± 25.48	4.25 ± 1.06	123.18 ± 66.75
	Е	0.86	1.73	0.00	67.77	0.13	5.27
		1.04	1.88	2.04	72.00	1.78	44.53
		0.68	2.04	2.13	38.22	2.40	31.35
	Avg.	0.86 ± 0.12	1.88 ± 0.10	1.39 ± 0.93	59.33 ± 14.07	1.44 ± 0.87	27.05 ± 14.52
Immersed in	А	2.24	1.08	4.00	20.07	3.47	42.73
5% NaOH		1.51	1.23	2.04	18.63	2.65	33.93
		1.26	1.41	4.44	40.69	2.30	26.61
	Avg.	1.67 ± 0.38	1.24 ± 0.11	3.50 ± 0.97	26.46 ± 9.48	2.81 ± 0.44	34.42 ± 5.54
	В	0.77	1.70	0.00	140.34	5.08	206.83
		0.73	1.46	2.00	94.44	5.90	95.49
		1.05	1.42	0.00	72.83	6.22	95.36
	Avg.	0.85 ± 0.13	1.53 ± 0.12	0.67 ± 0.89	102.54 ± 25.20	5.73 ± 0.44	132.56 ± 49.51
	С	0.31	2.62	0.00	121.41	10.64	321.01
		0.57	2.53	0.00	100.61	6.99	282.51
		0.52	2.57	0.00	148.64	5.11	103.06
	Avg.	0.47 ± 0.10	2.57 ± 0.03	0.00	123.55 ± 16.72	7.58 ± 2.04	235.53 ± 31.32
	D	0.60	2.07	2.04	79.98	6.15	87.66
		0.85	2.01	2.04	157.98	8.66	280.48
		0.85	1.99	0.00	152.44	0.74	276.45
	Avg.	0.68 ± 0.14	2.02 ± 0.03	1.36 ± 0.91	130.13 ± 33.43	5.18 ± 2.96	214.87 ± 84.80
	Е	1.74	1.35	1.96	97.39	3.16	87.53
		1.37	1.29	2.00	62.52	6.54	163.93
		1.45	1.21	2.04	43.16	0.00	3.05
	Avg.	1.52 ± 0.15	1.28 ± 0.05	2.00 ± 0.03	67.69 ± 19.8	3.23 ± 2.20	84.84 ± 54.52

Table 3. Chemical, physical, and mechanical properties of composites.

The water content of the composites is also shown in Figure 4a. The water content is higher following 5% NaOH than hot water treatment. The average water content of composites for all variations (samples A, B, C, D, and E) was 0.79% and 1.03% for composites modified by hot water immersion and 5% NaOH, respectively. According to [40], the alkalization process changes the fiber surface from hydrophilic to hydrophobic, so that the water content in the fiber decreases. However, hot water treatment seems to also cause changes in the fiber surface, in which it becomes hydrophobic and with an even lower water content than alkalized fibers. The water content in all samples complied with the SNI 06-3730-1995 standard. Wardhana et al., 2018 explained that the length of fiber soaking affects the composite's water content; the longer the soaking time, the lower the water content value [41].



Figure 4. Physical properties of water chestnut composite with modification by immersion in hot water and 5% NaOH: (a) water content, (b) density, and (c) thickness expansion.

3.2.2. Composite density

Composite density test results are shown in Table 3 and Figure 4b. The density of the composites was highest for sample C (40% e-glass fiber) and lowest for sample A (40% long water chestnut fiber): the highest density was found in composites with e-glass fiber composition. This is logical since e-glass has a density of 2.58 g/cm³ [32]. The lowest density of the hybrid composites was found in sample E (30%:10%:60%), indicating that greater volume fractions of long water chestnut fibers lead to lower composite density values; also, greater volume fractions of e-glass fiber lead to higher density values. The comparison between composite densities is clarified in Figure 4b. The 5% NaOH treatment produced a lower average density (i.e., 1.72 g/cm^3) than the hot water immersion (i.e., 1.92 g/cm^3). This density value is higher than the flax-e-glass-epoxy hybrid (1.30 g/cm³) and flax-epoxy fiber composites (1.245 g/cm³) in [33].

3.2.3. Composite thickness development

The lowest thickness expansion of the composites was obtained in sample C (Table 3). This might be caused by the e-glass fiber and polyester resin covering the composite tightly, making it difficult for water to enter the composite and decreasing thickness. Regarding the composites with modified 5% NaOH treatment, composite samples A and E have the highest thickness expansion value. Here, the composite absorbs more water due to the presence of the water chestnut fiber, which is hydrophilic.

The results of the composite thickness development test met the JIS A-5908-1994 standard, which is below 12%.

Figure 4c shows a comparison of the expansion of composite thickness based on treatment. The highest expansion in composite thickness was in sample A (3.50%), which is the composite without e-glass fiber. The lowest was in sample C (0%), the composite without water chestnut fiber. Meanwhile, the lowest thickness expansion of the hybrid composites was found in sample E; the higher the volume fraction of long water chestnut fibers, the higher the composite thickness expansion value. Also, the higher the e-glass fiber volume fraction, the lower the thickness expansion value.

3.2.4. Composite tensile strength

The results of the composite tensile strength test are shown in Table 3 and Figure 5. The tensile strength for all composite samples varied between 21.98 and 134.68 MPa. The highest composite tensile strength was found in sample D, both for immersion in hot water and 5% NaOH treatment: 134.68 and 130.13 MPa, respectively. The lowest tensile strength was obtained in sample A for both modifications (21.98 and 26.46 MPa). This shows that higher fiber volume fractions lead to greater voids occurring in the composite. These voids cause a decrease in the tensile strength of the composite [42]. The existence of cavities is the initial place for cracks, so composites that experience stress have low tensile strength values. The test results show that the higher the volume fraction of long water chestnut fibers, the lower the tensile strength value of the composite. At the same time, more e-glass fibers result in increased tensile strength. This is because the tensile strength of e-glass is higher than that of natural fibers [42]. In previous research, the flax-e-glass-epoxy hybrid composite obtained a tensile strength of 216.14 MPa, while the flax-epoxy fiber composite obtained a value of 81.68 MPa. These results indicate that the hybrid composite without e-glass (sample A) showed the lowest tensile strength.



Figure 5. Composite tensile strength following immersion in hot water and 5% NaOH treatment.

Figure 5 shows the comparison of tensile strength. The modification of 5% NaOH produced a higher average tensile strength (90.07 MPa) than immersion in hot water (86.71 MPa). Alkaline NaOH treatment causes an increase in the tensile strength of the composite [37] because this alkalization

causes the fiber's adhesion (binding) properties to be higher against other composite components. Based on the three variations in the volume fraction of long water chestnut fibers, i.e., 20%, 30%, and 40%, it can be said that the modification treatment of natural fibers fulfilled its purpose, providing a better bond between the fibers and the matrix, which ultimately improves the mechanical properties of the composite [15].

Tensile strength measurements are shown by the stress–strain relationship between the test material and the composite, shown in Figures 6 and 7. When the composite receives a load, the axial force is first received by the matrix; then, the force is applied to the fibers so that the larger the fraction of fiber volume, the greater the composite part that can withstand the workload. In this test, the composite is called flexible (ductile) if the plastic stress that occurs before breaking is higher than 5%, otherwise, the composite is called brittle [43].



Figure 6. Stress-strain tensile strength of modified composites with hot water immersion.

Figure 6 shows the stress-strain tensile strength of the composites modified by hot water immersion. Sample A, with a volume fraction of 40%:60% (long water chestnut fiber:polyester) immediately broke without experiencing deformation at an average strain of 0.82% with a maximum strength of 881.92 N. This composite can be said to be brittle and had the lowest maximum strength compared to other samples. Composite samples B, C, D, and E broke after experiencing an average

strain of 2.89%, 4.13%, 3.78%, and 2.51% with a maximum strength of 5,129.39, 7,258.50, 6,813.35, and 2,417.58 N, respectively. From these results, it can be seen that samples B, C, and D have stiff, firm, and strong properties since these composites produce quite high maximum strength values and do not break easily. Meanwhile, samples A and E have a smaller maximum strength, so it can be said that they are not strong and break easily.



Figure 7. Stress-strain tensile strength of modified composites with 5% NaOH treatment.

Figure 7 shows the stress-strain tensile strength of the composites with 5% NaOH modification. Composite sample A (40%:60%, long water chestnut fiber:polyester) immediately broke without experiencing deformation at an average strain of 1.00% with a maximum strength of 1,018.93 N. This composite can be said to be brittle because it has the lowest maximum strength compared to other samples. Composite samples B, C, D, and E broke after experiencing an average strain of 3.15%, 4.13%, 4.25%, and 2.52% with a maximum strength of 4,830.77, 7,258.50, 6,277.65, and 3,048.70 N, respectively. From these results, it can be seen that composite samples B, C, and D have stiff, firm, and strong properties because they produce quite high maximum strength values and do not break easily.

Meanwhile, samples A and E have a smaller maximum strength, so it can be said that the composite is not strong and also breaks easily.

3.2.5. Fracture cross-section morphology of composites after tensile strength test

Figures 8 and 9 show fractures in composite samples due to tensile strength measurements. Composite sample A experienced a single fracture and included a brittle fracture, which shows that the elasticity of composite sample A is not good. Composite sample B experienced a splitting fracture; this malleable and ductile material shows good elasticity. Composite sample B also showed fibers coming out of the matrix (fiber pull-out), which occurs when the bond between the fibers and the matrix is not optimal, so the matrix is unable to withstand the load at the end of the broken fiber. In this failure condition, the matrix and fibers are actually still able to withstand greater loads and stretch. Still, because the bond between the fibers and matrix fails, the composite fails earlier. The magnitude of the strain and stress when it fails is also lower. The fracture type for composite samples C and D was a ductile fracture, which shows good elasticity. For composite sample E, the type of fracture was a semi-brittle fracture, which shows poor elasticity. In this fracture, fiber also comes out of the matrix.



Figure 8. Fractures in the tensile strength test of modified composites immersed in hot water.



Figure 9. Fractures in the tensile strength test of modified composite with 5% NaOH treatment.

Several possibilities for this decrease in composite strength include the following: (1) there is trapped air (voids) in the composite. The greater the volume fraction of fiber as a composite reinforcement, the more voids there are in the composite. (2) Fiber distribution is uneven, so the strength of the resulting composite is also uneven at each point. (3) The resin and fiber bond do not blend perfectly. This will cause debonding (loose bonds between the fiber and the matrix) [43].

3.2.6. MoE and MoR of the composites

The MoE and MoR test results are shown in Table 3 and Figure 10. The highest MoE was obtained for composite sample C (40% e-glass fiber) at 7,580 MPa. The MoE results for composite samples D and B (e-glass fiber volume fractions of 30% and 20%) showed a slight difference. MoE was 430 MPa in the hot water immersion and 550 MPa in the 5% NaOH modification. From the test results of these three samples, it can be seen that the composites were able to resist the material undergoing deformation when force was applied. Meanwhile, composite samples A and E, with a volume fraction of 40% and 30% of long fiber water chestnut, had lower MoE values than the composites with hot water immersion or 5% NaOH modification, because this composite is not strong enough to withstand certain force.



Figure 10. MoE (a) and MoR (b) of modified composites by hot water immersion and 5% NaOH.

In Figure 10a, a MoE comparison can be seen. In the five samples tested, the composite with 5% NaOH modification had a higher MoE than the composite with hot water immersion. This means that the 5% NaOH modification can improve the mechanical properties of long water chestnut fibers used as composite reinforcement. In this study, the MoE value was greater than that of Setiyawan et al., [44], in which MoE was obtained at 702.7 kg/cm² or 68.911 MPa; research by [24] on hybrid composites with a volume fraction of 40% reinforcement obtained a MoE of 102 MPa.

In Table 3, it can be seen that MoR is the highest in sample C (40% e-glass fiber), namely 235.53 MPa. Furthermore, the successive values obtained from the MoR test results depend on the high-volume fraction of e-glass fibers in the composite sample. Composite samples D and B, with a volume fraction of 30% and 20% e-glass fiber modified by hot water immersion, had a MoR of 123.18 and 120.26 MPa, while 5% NaOH modification led to a MoR of 214.87 and 132.56 MPa. The resulting composites had a value high enough to determine fracture load. Meanwhile, composite samples E and A, with volume fractions of 10% and 0% of e-glass fiber, had the lowest MoR values with modification by immersion in hot water or 5% NaOH. This composite uses a lot of long water chestnut fiber reinforcement, so this composite breaks more easily than composites with e-glass fiber reinforcement.

In Figure 10b, a comparison of MoR values can be seen. In the five composite samples tested, the MoR value of the composite is determined by the amount of e-glass fiber used. The greater the volume fraction of e-glass fiber, the stronger the resulting composite, and the less easily broken it is. The composite with 5% NaOH modification had a higher MoR value than the composite with hot water immersion. This means that 5% NaOH modification can improve the mechanical properties of long water chestnut fibers used as composite reinforcement. Water chestnut fiber, a natural fiber, can absorb a smaller matrix than e-glass fiber, a synthetic fiber because natural fibers still contain water. So, the smaller the natural fiber volume fraction percentage, the higher the MoR and MoE values. The MoE value is directly proportional to the MoR value: the higher the composite fracture toughness value, the higher the elasticity.

3.2.7. Fracture cross-section morphology of composites after the bending test

Figures 11 and 12 show the composite bending test fracture results. Figure 11 shows the fracture of the composites following immersion in hot water. Samples A, B, and C experienced fiber pull-out fractures. This occurs when the matrix is unable to withstand the load that arises at the ends of the broken fibers, so the fibers come out of the matrix. In composite samples D and E, there are fractures by delamination or detachment of one lamina from another. Although polyester has excellent surface interaction with e-glass and water chestnut fibers, the volume fractions need to be balanced so that the hybrid composite does not experience delamination. This is related to the interfacial adhesion between the fiber reinforcement and the matrix [40].



Figure 11. Bending test fracture of modified composites by immersion in hot water.

Figure 12 shows fractures in the composite samples modified with 5% NaOH. The sample A experienced a single, brittle fracture, showing that its elasticity was not good. In sample B, there was delamination or detachment of one lamina from another. Samples C, D, and E showed ductile fractures, which translates to good elasticity; these also show fibers coming out of the matrix (fiber pull-out).



Figure 12. Bending test fracture of modified composite by 5% NaOH.

4. Conclusions

Modification of sedge fibers by soaking in hot (100 °C) water or 5% NaOH resulted in a decrease in water and lignin content. This caused an increase in cellulose content, which then increased density and tensile strength. In the composites, variations in the volume fraction of long fibers showed a correlation with water content, thickness swelling, density, and tensile strength. The highest tensile strength was obtained in sample D (10% volume fraction of long sedge fibers and 30% e-glass fibers), at 134.68 and 130.13 MPa for hot water immersion and 5% NaOH, respectively. The composites with 5% NaOH treatment had higher MoE and MoR than those immersed in hot water. The highest MoE and MoR values were recorded for sample C (40% e-glass fibers) at 7,580 and 235.53 MPa, respectively. Although the alkalization treatment of water chestnut fibers resulted in better water and lignin contents, density, and fiber surface interaction, the hot water treatment showed unexpected results. The decrease in water content was almost the same as in the alkalization treatment, while the fiber density was smaller and the tensile strength was greater. Further research is needed to better understand the mechanisms that occur in fibers with hot water treatment at different temperatures.

Use of AI tools declaration

The authors declare that we have not used Artificial Intelligence (AI) tools in the creation of this article.

Acknowledgments

Thank you to all who have assisted in this follow-up research, especially to the Institute of Research and Community Service at Lambung Mangkurat University which has provided funding through the PDWM Program.

Author contributions

The authors confirm their contribution to the paper as follows: study conception and design: Ninis Hadi Haryanti and Suryajaya; data collection: Khaipanurani, Adik Bahanawan and Setiawan Khoirul Himmi; analysis and interpretation of results: Khaipanurani, Tetti Novalina Manik, and Ninis Hadi Haryanti; draft manuscript preparation: Suryajaya. All authors reviewed the results and approved the final version of the manuscript.

Conflict of interest

The authors declare no conflict of interest.

References

- 1. Schwatz MM (1992) Composite Materials Handbook, New York: McGraw-Hill.
- Adnan N, Abral H, Dahlan H, et al. (2022) Identification of mechanical strength for mixture of thermoset polyester with thermoset vinyl ester due to bending load. *JMPM* 6: 19–25. https://doi.org/10.18196/jmpm.v6i1.14450
- 3. Mallick PK (2007) *Fiber-Reinforced Composites: Materials, Manufacturing, and Design*, Boca Raton: CRC Press Taylor & Francis Group. https://doi.org/10.1201/9781420005981
- Saba N, Jawaid M (2017) Epoxy resin based hybrid polymer composites, In: Thakur VK, Thakur MK, Pappu A, *Hybrid Polymer Composite Materials: Properties and Characterisation*, Amsterdam: Elsevier, 57–82. https://doi.org/10.1016/B978-0-08-100787-7.00003-2
- Nguyen KTQ, Navaratnam S, Mendis P, et al. (2020) Fire safety of composites in prefabricated buildings: From fibre reinforced polymer to textile reinforced concrete. *Compos B Eng* 187: 107815. https://doi.org/10.1016/j.compositesb.2020.107815
- 6. Alkbir MFM, Sapuan SM, Nuraini AA, et al. (2016) Fibre properties and crashworthiness parameters of natural fibre-reinforced composite structure: A literature review. *Compos Struct* 148: 59–73. https://doi.org/10.1016/j.compstruct.2016.01.098
- 7. Rosyidah K, Rohman T, Fitriani R (2018) Aktivasi antioksidan ekstrak metanol daun purun tikus (eleocharis dulcis). *JKPK* 3: 135–140. https://doi.org/10.20961/jkpk.v3i3.25626
- 8. Asikin S, Thamrin M (2012) Manfaat purun tikus (Eleocharis dulcis) pada ekosistem sawah rawa. *JLP* 31: 35–42. Available from: https://media.neliti.com/media/publications/30903-ID-manfaat-purun-tikus-eleocharis-dulcis-pada-ekosistem-sawah-rawa.pdf.
- 9. Firda AF, Fuad IS (2020) Pengaruh variasi perendaman beton purun tikus terhadap kuat tekan dan kuat lentur beton. JFM 9: 1–10. Available from: https://media.neliti.com/media/publications/456739-none-e00b882d.pdf.

- Prihatini NS, Krisdianto, Setyorini A, et al. (2011) Potensi purun tikus (eleocharis dulcis) sebagai biofilter. Proceedings environmental talk: Toward a better green living, 154–165. Available from: https://www.researchgate.net/publication/278968718_POTENSI_PURUN_TIKUS_Eleocharis_ dulcis_SEBAGAI_BIOFILTER.
- Suryajaya, Haryanti NH, Husain S, et al. (2020) Preliminary study of activated carbon from water chestnut (Eleocharis dulcis). J Phys Conf Ser 1572: 012053. https://doi.org/10.1088/1742-6596/1572/1/012053
- 12. Wardhana H, Haryanti NH (2017) The characteristics of purun tikus particle board cement board. *IOSR-JAC* 10: 1–4. https://doi.org/10.9790/0661-1001010104
- 13. Martinelli FRB, Pariz MG, de Andrade R, et al. (2024) Influence of drying temperature on coconut-fibers. *Sci Rep* 14: 6421. https://doi.org/10.1038/s41598-024-56596-z
- Misra S, Misra M, Tripathy SS, et al. (2002) The influence of chemical surface modification on the performance of sisal-polyester biocomposites. *Polym Compos* 23: 164–170. https://doi.org/10.1002/pc.10422
- 15. Witono K, Irawan YS, Soenoko R, et al. (2013) Pengaruh perlakuan alkali (NaOH) terhadap morfologi dan kekuatan tarik serat mendong. *JRM* 4: 227–234. Available from: https://rekayasamesin.ub.ac.id/index.php/rm/article/download/222/214.
- Kosjoko K (2015) Upaya peningkatan kualitas sifat mekanik komposit serat purun tikus (Eleocharis dulcis) bermatriks polyester dengan perlakuan NaOH. SNTTM XIV 1–5. Available from: http://eprints.ulm.ac.id/713/.
- Kosjoko K, Sonief AA, Sutikno D (2011) Pengaruh waktu perlakuan kalium permanganate (KMnO₄) terhadap sifat mekanik komposit serat purun tikus (Eleocharis dulcis). *JRMU* 2: 193– 198. Available from: https://www.neliti.com/id/publications/130838/pengaruh-waktu-perlakuankalium-permanganate-kmno4-terhadap-sifat-mekanik-kompos.
- 18. Haryanti NH, Suryajaya, Banowati L, et al. (2021) Modification of purun tikus (Eleocharis dulcis) as a natural fiber composite using KMnO₄ and NaOH. *SPEKTRA* 6: 37–47. https://doi.org/10.21009/SPEKTRA.061.05
- 19. Haryanti NH, Wardhana H, Suryajaya (2019) The Effect of NaOH immersion time to physical properties of *purun tikus (Eleocharis dulcis)* cement board composite. *IOP Conf Ser Mater Sci Eng* 599: 012013. https://doi.org/10.1088/1757-899X/599/1/012013
- 20. Purwanto EH, Iflah T, Aunillah A (2020) Pengaruh alkalisasi nib kakao terhadap kandungan kimia dan warna bubuk kakao, In: Herlinda S, *Prosiding Seminar Nasional Lahan Suboptimal*, 253–260.
 Available

https://conference.unsri.ac.id/index.php/lahansuboptimal/article/view/1931.

- Saidah A, Susilowati SE, Nofendri Y (2018) Pengaruh fraksi volume serat terhadap kekuatan mekanik komposit serat jerami padi epoxy dan serat jerami padi resin yukalac *JKEM* 5: 96–101. https://doi.org/10.21009/JKEM.5.2.7
- 22. Samal SK, Mohanty S, Nayak SK (2009) Banana/glass fiber-reinforced polypropylene hybrid composites: Fabrication and performance evaluation. *Polym-Plast Technol Eng* 48: 397–414. https://doi.org/10.1080/03602550902725407
- 23. Kumar KV, Moshi AAM, Rajadurai JS (2021) Mechanical property analysis on bamboo-glass fiber reinforced hybrid composite structures under different lamina orders. *Mater Today Proc* 45: 1620–1625. https://doi.org/10.1016/j.matpr.2020.08.423

- 24. Riyanto A, Irfa'I MA (2018) Pengaruh fraksi volume serat komposit hybrid berpenguat serat bambu acak dan serat e-glass anyam dengan resin polyester terhadap kekuatan bending. *JKM* 06: 55–60. Available from: https://ejournal.unesa.ac.id/index.php/jtm-unesa/article/view/25878/23717.
- 25. Gundara G (2017) Analisis sifat fisis dan mekanis komposit serat gelas berlapis. *Prosid Sem Nas Teknoka* 2: M17–M21. Available from: https://journal.uhamka.ac.id/index.php/teknoka/article/view/794.
- 26. Djunaedi T, Setiawan B (2018) Pengujian kekuatan tarik komposit variasi arah serat roving-resin polyester BQTN R157 yang diproduksi dengan metode vacuum bagging untuk aplikasi pesawat tanpa awak. *Semnastek UMJ Jakarta* 1–10. Available from: https://jurnal.umj.ac.id/index.php/semnastek/article/view/3528.
- Mamleev V, Bourbigot S, Yvon J (2007) Kinetic analysis of the thermal decomposition of cellulose: The main step of mass loss. J Anal Appl Pyrolysis 80: 151–165. https://doi.org/10.1016/j.jaap.2007.01.013
- 28. Maiti S, Purakayastha S, Ghosh B (2007) Thermal characterization of mustard straw and stalk in nitrogen at different heating rates. *Fuel* 86: 1513–1518. https://doi.org/10.1016/j.fuel.2006.11.016
- 29. Ezekiel N, Ndazi B, Nyahumwa C, et al. (2011) Effect of temperature and durations of heating on coir fibers. *Ind Crops Prod* 33: 638–643. https://doi.org/10.1016/j.indcrop.2010.12.030
- Mamungkas MI, Hendaryati H, Murjito M (2023) The effect of heating treatment on the tensile strength of pineapple leaf fiber composites using the vacuum infusion method. *J-Proteksion* 7: 69–72. https://doi.org/10.32528/jp.v7i2.8665
- Wicaksono ST, Laksana AH, Ardhyananta H (2019) Effect of calcium carbonate on the tensile and density properties of Kenaf/polyester hybrid composite. *IOP Conf Ser Mater Sci Eng* 546: 042019. https://doi.org/10.1088/1757-899X/546/4/042019
- EL-Wazery MS, EL-Elamy MI, Zoalfakar SH (2017) Mechanical properties of glass fiber reinforced polyester composites. *Int J Appl Sci Eng* 14: 121–131. https://doi.org/10.6703/IJASE.2017.14(3).121
- 33. Banowati L, Hartopo H, Octariyus G, et al. (2020) Analisis perbandingan kekuatan tarik komposit rami/epoksi dan hibrid rami-E-glass/epoksi. *INDEPT* 9: 80–89. Available from: https://jurnal.unnur.ac.id/index.php/indept/article/view/378.
- 34. ASTM International (2017) Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials. ASTM D790-00. Available from: https://www.astm.org/d0790-00.html.
- 35. ASTM International (2014) Standard test method for tensile properties of plastics. ASTM D638-14. Available from: https://www.astm.org/d0638-14.html.
- 36. Maryanti B, Sonief AA, Wahyudi S (2011) Pengaruh alkalisasi komposit serat kelapa poliester terhadap kekuatan tarik. *JRM* 2: 123–129. Available from: https://www.neliti.com/id/publications/128471/pengaruh-alkalisasi-komposit-serat-kelapa-polies ter-terhadap-kekuatan-tarik.
- Wang X, Chang L, Shi X, et al. (2019) Effect of hot-alkali treatment on the structure composition of jute fabrics and mechanical properties of laminated composites. *Materials* 12: 1386–1398. https://doi.org/10.3390/mal12091386

- Paramita O (2014) Pengaruh jenis air perendam terhadap kandungan vitamin C, serat, dan protein tepung mangga (Mangifera indica L.). *Jurnal Bahan Alam Terbarukan* 2: 1. https://doi.org/10.15294/jbat.v2i1.2912
- Hashim MY, Amin AM, Marwah OMF, et al. (2017) The effect of alkali treatment under various conditions on physical properties of kenaf fiber. J Phys Conf Ser 914: 1–15. https://doi.org/10.1088/1742-6596/914/1/012030
- 40. Bismarck A, Aranberri-Askargorta I, Springer J (2002) Surface characterization of flax, hemp and cellulose fibers; surface properties and the water uptake behavior. *Polym Compos* 23: 872–894. https://doi.org/10.1002/pc.10485
- Wardhana H, Haryanti NH (2018) Sifat fisik komposit papan semen berbahan serat purun tikus (eleocharis dulcis) dengan perlakuan perendaman NaOH. *Prosiding Seminar Nasional Tahunan* 10: 134–141. Available from: http://s2tekniksipil.ulm.ac.id/wp-content/uploads/2019/04/15.-Henry-Wardhana-dan-Ninis-Hadi-Haryanti.pdf.
- 42. Nurhidayat A, Susilo DD (2013) Pengaruh fraksi volume pada pembuatan komposit HDPE limbah-cantula. *Prog Pascasarj Tek Mesin Univ Sebel Maret Surakarta* 14: 1–70. Available from: https://core.ac.uk/download/pdf/33477114.pdf.
- 43. Gibson RF (1994) Principles of Composite Material Mechanics, New York: McGraw-Hill.
- 44. Setiyawan D, Respati SMB, Dzulfikar M (2020) Analisa kekuatan komposit sandwich karbon fiber dengan core styrofoam sebagai material pada model pesawat tanpa awak (uji tarik & uji bending). JIM 16: 1–5. Available from: https://jurnal.unwahas.ac.id/MOMENTUM/article/view/3345.



© 2024 the Author(s), licensee AIMS Press. This is an open access article distributed under the terms of the Creative Commons Attribution License (https://creativecommons.org/licenses/by/4.0)