The Effect of Boiling Time of Wulung Bamboo Fiber (*Gigantochloa Atroviolacea*) in NaOH Solution on Tensile Strength of Epoxy Matrixed Composites

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*The purpose of this study was to determine the effect of immersion time of NaOH at a temperature of 60-70 °C in wulung bamboo fibers on the tensile strength of the epoxy matrix composites. Wulung bamboo fibers (*Gigantichloa Atroviolacea*) have the potential to be used as reinforced in composites because they have high strength and are widely grown in Indonesia. In this study, bamboo wulung fiber reinforcement was used without treatment and with variations, soaking time 30 minutes, 60 minutes, and 90 minutes in 6% NaOH solution with a temperature of 60-70 °C. The composite manufacturing method used was vacuum assisted resin infusion and the standard composite specimen used as a tensile test was ASTM D638-01. The results showed that the longer the immersion time resulted in a rougher topography of the fiber surface which caused the composite tensile strength to increase due to the better mechanical interlocking between the fibers and the epoxy resin matrix. The wettability results on the fibers also show that the wetness between the fibers and the epoxy resin matrix is getting better, which is shown from the droplet contact angle on the resulting fibers. The highest composite tensile strength was obtained at 90 minutes immersion time of 169.765 MPa followed by immersion time of 60 minutes of 123.2 MPa, immersion time of 5 minutes of 81.7 MPa and without treatment of 71.6 MPa. Based on the results of the macro photo shows that the treatment given will reduce the failure of the pull out and debonding defects on the composite fracture.*

Keywords : Bamboo Wulung Fiber, Temperature, Immersion, Tensile Strength, Composite, Mechanical Interlocking.

1. INTRODUCTION

In modern times like today, of course, technological developments will continue to develop over time. In today's era, many scientists are researching or making new breakthroughs on environmentally friendly innovations, where scientists take advantage of the natural wealth on earth which is then transformed into something of more value. One of them is the development of "composites" in the material field. Composite itself is a material that is formed from a combination of two or more materials, where the mechanical properties of the forming materials are different. Due to the different forming characteristics, a new material will be produced, namely a composite that has different mechanical properties and characteristics from the forming materials [1]

Unlimited natural fiber reinforced biocomposite materials have begun to be developed, one of which is natural fibers such as jute, sisal, hemp, bamboo and banana which have been applied to industries, namely dashboards, car bodies, aircraft industry and other interior devices [2].

With the abundance of natural resources in Indonesia, of course it will be very beneficial if we can make the most of our natural resources. Indonesia itself is famous for its abundant natural wealth, one of which is bamboo. Bamboo is a plant that is often found and thrives in all parts of Indonesia. Bamboo itself has a faster growing time and a lower price than wood. Indonesia has 76 species of bamboo from 17 genera [3]

Of the many types of bamboo in Indonesia, the writer chooses to use bamboo wulung (*Gigantochloa atroviolacea*). This bamboo is commonly called black bamboo because the color of the stem is blackish or dark purple. The trunk is upright with a height of 20 m. The length of the sections is 40-50 cm, the thickness of the reed walls is 8 mm, so that the moisture content is not too high [4].

Wulung bamboo grows a lot in Java and its surroundings and is also widely used for various building materials as well as various kinds of crafts. The use of black bamboo by Indonesians is high because it is considered to have a versatile function, easy to obtain and at an affordable price .

Based on the explanation above, the waulung bamboo fiber has the potential to be used as a composite reinforcing material. The use of bamboo wulung as a composite reinforcing material will be an added value from the utilization of wulung bamboo.

Natural fibers themselves are hydrophilic, causing the adhesion force between the fiber and the low-value matrix. Therefore, to optimize the properties of cellulose and to increase the adhesion force between the fibers and the polymer matrix, treatment of the fibers is needed [5]. This treatment can be in the form of chemical treatment or physical treatment.

In this study, the NaOH solution was extracted using boiling assistance so that its chemical properties could be perfectly integrated [6]. The increase in mechanical properties of the treated fiber reinforced composites indicated the fact that there was an improvement in the adhesion characteristics of the fiber surface by repairing the natural defects and the surface topography of the fibers became coarse [8]. The purpose of this study was to determine the effect of boiling time in NaOH solution on bamboo fiber wulung (*Gigantochloa atroviolacea*) on the matrixed tensile strength of epoxy.

2. MATERIALS AND METHODS

The research method used in this research is (True Experimental Research) and the processing of bamboo wulung fibers can be seen in Figure 1. The diagram of the fiber processing in NaOH solution at temperatures of 60-70 °C

Figure 1. Flow chart of fiber processing.

The preparation of materials and tools is shown in table 1 below:

Table 1. The preparation of materials and tools

Parameters	Information
Boamboo age	± 2 years
Fiber mass (Totally)	2 gram
Fiber mass (fraction)	25%
Threatment	Without treatment, 30 minutes, 60 minutes, and 90 minutes with 6% NaOH concentration at temp. 60-700 celcius.
Oven Drying Time	15 Minutes
fiber arrangement	<i>Continuos</i>
The ratio of epoxy resin A to hardener	2:01

This research method uses vacuum assisted resin infusion which in the manufacture of composite

manufacturing as shown in Figure 2. The epoxy resin is flowed into the composite mold by utilizing a constant difference in pressure due to the air in the mold being sucked in by the vacuum infusion pump. The resin that has passed through the mold will be accommodated by the resin trap.

Figure 1. Composite manufacture scheme

3. TENSILE TESTS

The composite test specimen used is in accordance with the ASTM D638-01 standard for tensile testing[7]. The dimensions of the specimens are shown in Figure 3. in millimeters (mm).

Figure 3. D638-01 specimen shape

The tensile test will be carried out on three composite specimens and the stress and strain values will be obtained from the composite specimens. The voltage is formulated like the equation below:

$$\sigma = F / A \quad (1)$$

Information :

σ = Normal stress due to static tensile load (N / mm²)

F = tensile load (N)

Ao = sectional area of the specimen initial (mm²)

The strain is formulated like the equation below:

$$\varepsilon = \Delta L / L \quad (2)$$

Information :

ε = Strain due to static tensile load

L = Change in specimen length due to tensile load (mm)

Lo = Length of initial specimen (mm)

$\Delta L = L - L_0$

3. RESULTS

3.1 Data of Tensile Test for Bamboo Wulung Single Fiber

Figure 4. Graph of the average strength testing of single fiber bamboo wulung

Figure 4 shown that the average tensile strength of the wulung bamboo fibers without treatment is 183 MPa, followed by the tensile strength of the wulung bamboo fibers with a 30-minute soaking time of 165 MPa, then the tensile strength of the wulung bamboo fibers with 60 minutes of soaking results amounted to 141 MPa and the immersion time was 90 minutes which was 110 MPa. From the graph, it can be concluded that the higher the immersion time at 6% NaOH concentration, the less tensile strength of the bamboo wulung fibers will be.

The long process of soaking NaOH at a temperature of 60-70 °C removes impurities on the bamboo wulung fibers and degrades the chemical content of wulung bamboo fibers, namely wax, cellulose and lignin. The duration of soaking NaOH at a temperature of 60-70 °C aims to separate lignin from wulung bamboo fibers which make the fibers coarse and are replaced by epoxy matrices in composites in order to produce better mechanical interlocking with matrices[8]. That the data obtained is in accordance with the theoretical basis which states that the tensile strength will decrease along with the alkalization treatment due to the loss of lignin and hemicellulose which causes the fiber to experience a decrease in the tensile strength of a single fiber[9].

The reduced lignin can be proven from the FTIR test results in Figure 6.shows a graph of the FTIR test results and Table 3.That lignin has a wavelength which is indicated by a peak in the range 1500 cm⁻¹ - 1600 cm⁻¹ limited by the red line in Figure 5. with the aromatic functional group C = C [10].

Figure 5. FTIR test chart of bamboo wulung fiber

Table 3. The resulting ring content $C = C$

Threatment	Wave Num- ber (cm^{-1})	Intensity (%T)
UT	1531.374	75.90589
30M	1537.161	73.58269
60M	1535.232	71.36645
90M	1531.374	69.23133

Based on the data obtained from the FTIR test results in Figure 5. and Table 3. It can be seen that there is lignin in bamboo wulung fibers without treatment and with immersion treatment for 30 minutes, 60 minutes and 90 minutes. This shows that there is still an aromatic ring wave number $C = C$ in the range 1500 cm^{-1} - 1600 cm^{-1} . There is a reduction in intensity (percent transmittance) in the long treatment of soaking NaOH at a temperature of $60-70^\circ\text{C}$. This is because the bamboo fibers that are subjected to lignin treatment experience extraction which reduces the lignin so that the intensity of the lignin decreases[11].

3.2 Data on the Tensile Strength of Wulung Bamboo Fiber Composites

Figure 6. Graph of the tensile strength of the composite bamboo fiber reinforced wulung **Figure 7.** Graph of average tensile strength of bamboo fiber reinforced composites

Figure 6 shown that the composite bamboo fiber reinforced wulung works as reinforcement in the composite because the tensile strength value without treatment produced exceeds the tensile strength of the binder, namely epoxy and with higher NaOH treatment compared to epoxy specimens.

In Figure 7, it can be seen that the tensile strength of the bamboo wulung fiber reinforced composites has increased at 30 minutes, 60 minutes and 90 minutes. The average tensile strength of the composite without NaOH treatment resulted in a value of 73.9 MPa. Then with the treatment of 30 minutes immersion yields a value of 93.7 MPa and treatment of 60 minutes and 90 minutes of immersion yields values of 118.3 MPa and 152.3 MPa. Whereas the untreated wulung bamboo fibers still have high lignin and hemicellulose on the fiber surface and on the bonds between the fibers. This will cause the epoxy resin to be unable to bind and seep all parts of the bamboo wulung fiber. However, in the treatment, the fiber surface becomes rougher and stiffer.

This is caused by lignin and hemicellulose which begin to degrade, making the surface of these fibers rougher. The rougher the surface on the fiber, the better mechanical interlocking with the matrix[12]. This is similar to the results of previous studies using bagasse fibers that the treatment using a long soaking time of NaOH solution at a temperature of 60-70 °C can increase the good adhesion between bagasse fiber and the matrix due to the removal of hemicellulose and lignin that holds the matrix in the fiber [13].

3.4 Stress-Strain Result Data Composite Tensile Test

Figure 8. Graph of the composite tensile stress - strain test

Figure 8 shows a graph of the stress-strain from the results of the composite tensile test. The highest composite tensile strength with bamboo wulung fiber reinforcement with 90 minutes immersion time is 169,765 MPa. Followed by the highest composite tensile strength, namely the treatment for 60 minutes of immersion, which was 122,524 MPa, then the composite tensile strength at 30 minutes of immersion was 100,159 MPa and continued with the weakest composite tensile strength without treatment, which was 66,530 MPa.

The untreated wulung bamboo fiber composite had a value of 66,530 MPa and a strain value of 5.446%. Then it produces a modulus of elasticity of 1.203 GPa. Wulung bamboo fiber composites in the 30 minute immersion treatment had a stress value of 122,524 MPa and a strain value of 5,976%. Then it produces a modulus of elasticity of 1,676 GPa. Furthermore, the wulung bamboo fiber composites in the treatment for 60 minutes of immersion resulted in a stress of 122.524 MPa and a strain of 6.82%. This results in a modulus of elasticity of 1.796 GPa. Then in the bamboo fiber composite wulung on the treatment of 90 minutes soaking time had a stress of 169,765 MPa and a strain of 7,814%. Then it produces a modulus of elasticity of 2.172 GPa.

3.5 Composite Fault Analysis Using Macroscopic Photos

In general, fractures are divided into two, namely ductile fracture and brittle fracture. Macro ductile fractures are characterized by the cross-sectional edge of the fracture forming an angle of 45 ° and the middle part in the form of fibers. However, the brittle fracture is characterized by a 90 ° fracture and a rough shape. And at macro level, a fracture can be said to be brittle fracture, but micro-brittle fracture can be cleavage or intergranular [14]. This is the fracture in the bamboo wulung fiber without treatment or treatment for long immersion of NaOH at a temperature of 60-70 °C is called a brittle fracture which is shown by the macro photo of each specimen.

Macroscopic photos of fracture composites are used to observe defects that occur due to tensile loads on the composite. The defects that often occur in natural fiber reinforced composites are fiber pull out and debonding. Fiber pull out is the removal of natural fibers from the matrix as a result of the fiber's low ability to bind with matrix[15]. Meanwhile, debonding is a mechanism for the release of bonds between constituent materials when the composite specimen receives a load so that the matrix and fibers are peeled off. Debonding causes the matrix to not be able to distribute the load on the bamboo wulung fibers when receiving tensile loads. Debonding is due to low adhesion between the composites[16]

Figure 9. Macroscopic fracture of composite fracture reinforced with bamboo fiber wulung (A, B) without treatment; (C, D) 30M; (E, F) 60M; and (G, H) 90M

In Figure 9A and Figure 9B, it can be seen that there are a lot of pull out defects, this is because the untreated fiber still has a natural matrix which affects the adhesion between the fiber and the epoxy matrix. This specimen has a pull out defect of about 5 mm. This specimen also shows debonding defects. Debonding is due to the low adhesion between the composites [17]. The pull out and debonding defects cause the tensile strength of this composite specimen to be 78,748 MPa.

In Figures 9C and 9D shows the fracture of the composite reinforced bamboo wulung fibers with a 30-minute soaking time of NaOH at a temperature of 60-70 °C. The treatment given is able to modify the fiber surface so that the adhesion between the fibers with the epoxy matrix is getting better. This is indicated by the reduction in the length of the pull out on the specimen by about 3mm. Debonding also occurs due to the weak adhesion between the wulung bamboo fibers and the epoxy matrix. The maximum tensile strength in this specimen is 100.159 MPa.

Figures 9E and 9F show the fracture of the composite reinforced bamboo wulung fibers with a 60-minute immersion period of NaOH at a temperature of 60-70 °C. The pull out defects become fewer and shorter. The maximum tensile strength of this composite is 123.096 MPa.

Figures 9G and 9F show the fracture of the composite with 90 minutes of immersion in NaOH at a temperature of 60-70 °C. The resulting pull out defect is slight. Debonding is also visible in this composite, only slightly visible due to the bond between the matrix and the strong fibers. The maximum tensile strength in this specimen is 169,765 MPa. The composite specimens in the tensile test in this study experienced fractures in the gage length area. This is shown in Figure 10 (a) to Figure 10 (d).

Figure 10. Fracture of wulung bamboo fiber composites (a) TP, (b) 30M, (c) 60M, (d) 90M

3.6 Wettability Test Results

Figure 11 shows that the wettability properties of the untreated fiber and the long immersion treatment resulted

in different angles to the epoxy resin droplets on the surface of the bamboo wulung fiber. Whereas the contact angle generated from each fiber is used to measure the surface free energy and depends on the chemical structure on the fiber surface, the smaller the contact angle, the greater the part of the active central free energy, this will later function as an increase in the adhesion properties of the fiber. the contact surface. The result (a) shows that the resulting angle is 59° , that there is still a lot of dirt on the surface of the fiber, which prevents the fiber from getting wet. In Figures (b) and (c), the results show that the angle contact is smaller than the untreated fibers at 45° and 37° , this means that the immersion time of NaOH has a better impact on the adhesion properties of the fiber, so that the contact angle of the droplet epoxy droplets the resulting smaller. Whereas in Figure (d) the resulting contact angle is the smallest of all fiber surfaces, which is 33° , showing that the long immersion time makes the fiber wet. It can be seen that the smaller the contact angle the better the wettability, so that the matrix as a fiber adhesive medium must have the ability to cover the surface area of the fiber optimally. It can be concluded that the longer the immersion in the fibers, the smaller the resulting contact angle. This is because the surface of the fiber becomes rougher, stiffer and porous, which makes the adhesion of the fiber to the droplet liquid even greater. The increased adhesion shown from the slackness of the contact angle will make the bond between matrix and fiber better[8].

Figure 11. Contact Angle between bamboo wulung fibers and resin droplets (a) without treatment, (b) 30M, (c) 60M, and (d) 90M

4. CONCLUSION

From the results of the research that has been carried out, it can be concluded that the average tensile strength of the wulung bamboo fiber composites when sorted from lowest to highest are: wulung bamboo fiber reinforced composites without NaOH immersion treatment of 73.912 MPa; 30 minutes immersion treatment of 93.729 MPa; treatment of 60 minutes immersion of 118.264 MPa; and 90 minutes immersion time of 152.394 MPa. This shows that the longer the immersion at 6% NaOH concentration, the higher the composite tensile strength because NaOH can change the fiber surface topography to be coarser which makes the mechanical interlocking between the fiber and the epoxy resin matrix better and better adhesion between the fibers. the inner fiber surface optimally absorbs the epoxy resin on the fiber surface.

REFERENCES

- [1] SILVA F DE A, CHAWLA N, FILHO RD DE T. Tensile behavior of high performance natural (sisal) fibers. *Compos Sci Technol* 2008. <https://doi.org/10.1016/j.compscitech.2008.10.001>.
- [2] SAXENA M, MORCHHALE RK, ASOKAN P, PRASAD BK. Plant fiber - Industrial waste reinforced polymer composites as a potential wood substitute material. *J Compos Mater* 2008. <https://doi.org/10.1177/0021998307087014>.
- [3] WONG KJ, YOUSIF BF, LOW KO. The effects of alkali treatment on the interfacial adhesion of bamboo fibres. *Proc Inst Mech Eng Part L J Mater Des Appl* 2010. <https://doi.org/10.1243/14644207JMDA304>.
- [4] LIU D, SONG J, ANDERSON DP, CHANG PR, HUA Y. Bamboo fiber and its reinforced composites: Structure and properties. *Cellulose* 2012. <https://doi.org/10.1007/s10570-012-9741-1>.
- [5] PANDIATMI P, DYAH E, WATONI S. Polyester Composite Bending Strength Optimization Strengthened with Coastal Cottonwood Tree Bark Fiber and Fly Ash Filler. *IOP Conf. Ser. Mater. Sci. Eng.*, 2019. <https://doi.org/10.1088/1757-899X/494/1/012010>.
- [6] ALAWAR A, HAMED AM, AL-KAABI K. Characterization of treated date palm tree fiber as composite

- reinforcement. *Compos Part B Eng* 2009. <https://doi.org/10.1016/j.compositesb.2009.04.018>.
- [7] ASTM. *Astm D3039/D3039M*. Annu B ASTM Stand 2014. <https://doi.org/10.1520/D3039>.
- [8] PALUNGAN MB, SOENOKO R, GAPSARI F. The effect of king pineapple leaf fiber (*Agave Cantala Roxb*) fumigated toward the fiber wettability and the matrix epoxy interlocking ability. *EnvironmentAsia* 2019. <https://doi.org/10.14453/ea.2019.50>.
- [9] KORONIS G, SILVA A, FONTUL M. Green composites: A review of adequate materials for automotive applications. *Compos Part B Eng* 2013. <https://doi.org/10.1016/j.compositesb.2012.07.004>.
- [10] REDDY KO, ZHANG J, ZHANG J, RAJULU AV. Preparation and properties of self-reinforced cellulose composite films from *Agave* microfibrils using an ionic liquid. *Carbohydr Polym* 2014. <https://doi.org/10.1016/j.carbpol.2014.08.054>.
- [11] SURYANTO H, MARSYAHYO E, IRAWAN YS, SOENOKO R. Morphology, Structure, and Mechanical Properties of Natural Cellulose Fiber from Mendong Grass (*Fimbristylis globulosa*). *J Nat Fibers* 2014. <https://doi.org/10.1080/15440478.2013.879087>.
- [12] SENA NETO AR, ARAUJO MAM, SOUZA F V.D., MATTOSO LHC, MARCONCINI JM. Characterization and comparative evaluation of thermal, structural, chemical, mechanical and morphological properties of six pineapple leaf fiber varieties for use in composites. *Ind Crops Prod* 2013. <https://doi.org/10.1016/j.indcrop.2012.08.001>.
- [13] PARRE A, KARTHIKEYAN B, BALAJI A, UDHAYASANKAR R. Investigation of chemical, thermal and morphological properties of untreated and NaOH treated banana fiber. *Mater. Today Proc.*, 2020. <https://doi.org/10.1016/j.matpr.2019.06.655>.
- [14] JI M, LI F, LI J, LI J, ZHANG C, SUN K, et al. Enhanced mechanical properties, water resistance, thermal stability, and biodegradation of the starch-sisal fibre composites with various fillers. *Mater Des* 2021;198. <https://doi.org/10.1016/j.matdes.2020.109373>.
- [15] MYLSAMY B, PALANIAPPAN SK, Subramani SP, Pal SK, Sethuraman B. Innovative characterization and mechanical properties of natural cellulosic *Coccinia Indica* fiber and its composites. *Mater Test* 2020;62. <https://doi.org/10.3139/120.111451>.
- [16] BHUVANESHWARAN M, SUBRAMANI SP, PALANIAPPAN SK, PAL SK, BALU S. Natural Cellulosic Fiber from *Coccinia Indica* Stem for Polymer Composites: Extraction and Characterization. *J Nat Fibers* 2021;18. <https://doi.org/10.1080/15440478.2019.1642826>.
- [17] ZHAO FM, TAKEDA N. Effect of interfacial adhesion and statistical fiber strength on tensile strength of unidirectional glass fiber/epoxy composites. Part I: Experiment results. *Compos Part A Appl Sci Manuf* 2000;31. [https://doi.org/10.1016/S1359-835X\(00\)00085-3](https://doi.org/10.1016/S1359-835X(00)00085-3).