Investigation of characteristics of bamboo fiber for composite structures

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Abstract. Bamboo fibers have been used for reinforcements in structural composites because of their environmental friendly properties. The purpose of this study was to investigate the characteristics of three types of bamboo, namely Tali bamboo, Gombong bamboo and Haur Hejo bamboo to meet the requirements for a reinforcement in structural composites. The three types of bamboo fibers were alkaline treated by varying the concentrations of NaOH (4% and 6%), and immersion times (1, 2, and 3 hours). The alkaline pre-treatment was conducted at the temperature of 120°C and the pressure of 0.5 MPa using a steam explosion method. The treated bamboo fibers were characterized by FTIR and measured their tensile strengths. The tensile test results showed that the tensile strengths of Tali, Gombong, and Haur Hejo bamboos were 710 MPa, 418 MPa, and 457 MPa respectively. Whereas, the FTIR analysis indicated that there was still a peak of lignin with small intensity marked by aromatic C = C (alkyne) at the wavelength of 1500-2600 cm⁻¹ for all types of bamboo. There was also a peak of cellulose with an increase in the intensity marked by the aromatic group C = H (alkene) at the wavelength of 1900-2400.

1. Introduction
Sources of producing polymer fibers can be divided into 4 groups, namely 1) Animal fibers; 2) Synthetic polymer fibers made from petroleum fractions; 3) Ceramics/silica/glass fibers, and 4) Plant fibers. Natural polymer fibers refer to fibers obtained from extraction from plants such as hemp, straw, wood, sugar cane and bamboo, kenaf, hemp, oil palm empty bunches, sisal, coir, water hyacinth, banana fiber [1]. In addition to renewable sources, natural fiber is also biodegradable, abundant with fast and inexpensive production cycles and have the potential to replace synthetic polymer fibers as well as solutions to environmental impacts [2-8].

Bamboo is a source of natural fiber with abundant availability with limited application to meet needs such as houses, walls, ornaments, traditional equipment and even consumed in the form of bamboo shoots. The economic value and function of bamboo can be increased through the diversification of
natural polymer fiber products into bio-composites [3,5] for various lightweight structure applications [9], sound vibration dampers [10-12], biofilters [13], and thermal [14]. From various research and development of this diversification potential it is very prospective because of the superior mechanical properties of bamboo, namely low density i.e. 0.8-1.5 g/cm$^3$, tensile strength of 500-575 MPa, high elastic modulus of 27-40 GPa [11,15].

Bamboo fiber has a chemical composition of 55% fiber consisting of 34.5% cellulose and 2.5% hemicellulose, 26% lignin and the remaining volatile content and is hydrophobic. The bond of lignin with cellulose (lignocellulose) and hemicellulose and other extractive compounds forms a structure blocking the adhesion of cellulose polymer fibers to the matrix in producing composites. Separation of lignin-cellulose (lignocellulose) and hemicellulose and other extractive compounds was carried out by dissolving bamboo fiber NaOH solution with steam explosion method [16,17], and kenaf fiber into NaOH solution [18]. The results of fiber extraction can increase adhesion [19], also the bond of adhesion between fibers and matrices [20]. Tensile strength and composite modulus of fibers extracted by steam explosion increased by 15-30% with polypropylene matrix [16,17]. In other words, critical to quality (CTQ) natural fiber composites are the tensile strength of a single polymer fiber and adhesion to the matrix, either in the form of a thermoplastic matrix and/or thermosetting.

The extraction process of lignocellulose with NaOH solution is known as an alkaliization process or extraction process wherein the lignocellulose chain breaks into cellulose and lignin [16-20] as shown in Figure 1. Based on figure 1, OH$^-$ (results of dissociation of NaOH into Na$^+$ and OH$^-$) reacts with the H group on lignin, then forms H$_2$O. Cluster O forms free radicals and reacts with C to form epoxy rings (C-O-C). The reaction produces two separate benzene rings, where each ring has a reactive O group. This reactive group O reacts with Na$^+$ and dissolves in alkaline solutions so that lignin is removed when rinsed [16-21].

![Figure 1. Lignocellulose reaction in NaOH solution [3,21].](image)

Based on figure 1, it can be seen that lignin has a functional group OH, C-O-C, C-C, C = aromatic, CH aliphatic, CH$_2$, while cellulose OH and C = O. The effectiveness of the lignin extraction process from lignocellulose with NaOH solution is influenced by the concentration of NaOH solution, pressure and extraction time, and diameter of bamboo fiber. The effect of the extraction process with NaOH solution can be detected by FTIR spectrometry [22].

This FTIR spectrometry characterizes organic compounds based on their constituent functional groups, where each functional group of an organic molecule has energy and rotation levels with the value of wave numbers absorbed by the bonds of a different functional group. A typical infrared scan is generated in the mid infrared region of the light spectrum. The mid-infrared region is 400-4000 cm$^{-1}$ wave number, which is equal to the wavelength 2.5 to 25 microns. The correlation between wave numbers indicated by peaks with functional groups of natural fiber compounds are shown in table 1.
Table 1. Correlation of wave numbers to functional groups on natural fibers [23].

| Sources of fiber       | Functional groups. | -OH  | C-O-C | C-C  | C-H  | C-H2 | C=C  | C=C ring aromatic |
|------------------------|--------------------|------|-------|------|------|------|------|------------------|
| Bamboo [24]            | Weave number- WOT  | 1590 | 1161  | 1027 | 896  | 2900 | 1420 | 1200-1300        |
|                        | Weave number- WT   |      |       |      |      |      |      | 1238.97          |
| Ketandan sawit [10]    | Weave number- WOT  | 1593.46 | 1161.32 | 1027.49 | 895.57 | 2918.62 | 1420.32 | 1200-1300        |
|                        | Weave number- WT   | 1592.74 | 1161.32 | 1028.69 | 895.72 | 2918.62 | 1420.32 | 1200-1300        |
|                        |                    | 3322.84 | 1028.69 | 895.57 | 2918.62 | 1420.32 | 1200-1300        |
| [12]                   | Weave number- WOT  | 1627 | 1250  |      |      |      |      | 1319             |
|                        | Weave number- WT   |      |       |      |      |      |      | 1735             |

Note: WOT: without extraction; WT: with extraction.

To calculate the tensile strength of a single polymer cellulose fiber, which determines the tensile strength of the resulting composite, it is calculated using a tex unit [12],

\[
1 \text{ tex} = \frac{1000 \times W}{0.05} \quad (1)
\]

Where W is the weight of the test fiber sample in milligrams (W). Furthermore, the calculation of tensile strength of fiber is expressed as gram/tex, where the value of a is in milligrams,

\[
(\text{gram/tex}) = \frac{a}{W \ (\text{in unit tex})} \quad (2)
\]

The gram/tex value is converted to gram/denier, where 1 denier = 1/9 tex, the calculation of tensile strength (\(\sigma\)) is stated [12],

\[
\sigma = 130 \times \frac{\text{gram}}{\text{denier}} \ (MPa) \quad (3)
\]

The effectiveness of the extraction process of lignin and hemicellulose from lignocellulose bamboo fiber polymer with NaOH solution can be measured by FTIR spectrometry where the functional groups are identified [22-25], meanwhile the morphology of fiber can be seen by SEM results [12,26,27].

2. Experiment procedure

2.1. Bamboo fiber preparation

The stages of the extraction process and fiber characterization are shown in figure 2. Fiber is extracted from 3 types of bamboo, namely bamboo Tali, Haur Hejo bamboo and Gombong bamboo. The three types of bamboo samples are made of strips size 300-500 x 100 x 50 mm³ (l x w x t). The fiber extraction process was carried out in an autoclave at a temperature of 120°C with a pressure of 0.5 MPa with a variation of 4 and 6% NaOH solution, and extraction time of 1, 2, and 3 hours. Furthermore, the bamboo
strip sample was neutralized by using HCl and rinsed the water at a temperature of 90-95°C, and dried it in an oven at a temperature of 105°C for 4 hours.

Characterization of single fibers includes tensile strength, lignin and cellulose levels, morphology and identification of functional groups of wavenumber-based fiber compounds using FTIR spectrometry. Data compilation, analysis and evaluation related to the influence of extraction process parameters, lignin and cellulose levels and morphology are related to tensile strength. The functional groups are related to the levels of lignin and cellulose which have an impact on tensile strength. Based on the concept of measurement, analysis and evaluation, improvement can be determined key process parameters that produce maximum tensile strength and answered whether bamboo fibers have good prospects to replace synthetic fibers. Flowchart of bamboo extraction process and characterization it can be seen in figure 2.

![Flowchart of bamboo extraction process and characterization](image)

**Figure 2.** Flow chart of bamboo extraction process and its characterization.

### 2.2. Testing the pull of single bamboo fibers

The single tensile strength of bamboo polymer fibers is a determinant of the composite strength of the three types of bamboo fibers. Preparation of tensile test samples consisted of 150-200 mm long fiber bundles and 5 mm band width, and fiber weight 400-600 mg/bundle. The two ends of the fiber bundle are given adhesive paper so that the distance between the two adhesive papers is 50 mm. The tensile test sample as shown in figure 3 is mounted on a Tenso Lab-168 E tensile testing machine, with a cross head speed of 1.3 mm/minute to break up within 20 + 3 seconds according to ASTM D3379 or Indonesian National Standard (SNI) 08-1112-1989.

![Illustration of preparation tensile strength testing](image)

**Figure 3.** Illustration of preparation tensile strength testing: (a) single fiber; (b) mounting single fiber [28].
2.3. Testing for hemicellulose, cellulose and lignin
Tests for lignin levels using the SNI 0492: 2008 procedure, while for cellulose and hemicellulose levels using the SNI 0444: 2009 procedure.

2.4. FTIR testing
FTIR spectrometer bonds of bamboo fibers of Gombong (Gigantochloa pseudoarundinacea), Haur Hejo (Bambusa tuloides), and Tali (Gigantochloa apus) by using the standard of ASTM E1252. FTIR's specimen is a pellet consisting of a mixture of cellulose and KBr. FTIR testing was performed by using Shimadzu IRAffinity-1. A typical infrared scan is generated in the mid infrared region 400-4000 cm⁻¹ wave number, which is equal to the wavelength 2.5 to 25 microns of the light spectrum.

2.5. SEM testing
To identify the effect of fiber extraction process on morphology SEM testing was carried out using the JEOL JSM-6360, 15 kV with magnifying of 1,500 times.

Compilation of process parameters and test results data such as tensile strength, levels of lignin and cellulose, identification of functional groups and morphology then analysed and evaluated which answers whether bamboo fibers can replace synthetic fibers.

3. Results and discussion

3.1. Effect NaOH concentration and extraction time to tensile strength of single fiber
Data compilation of tensile testing results from Tali, Haur Hejo and Gombong bambooss are presented in table 2.

Table 2. Effect of fiber extraction process parameters on tensile strength of single fiber.

| Type of Bamboo | Process Parameters | Test result of Tensile strength of single fiber (MPa) | Average Tensile strength (MPa) |
|----------------|-------------------|-----------------------------------------------|-------------------------------|
|                | Autoclave (T=120°C, and P= 0.5 MPa) | | |
|                | Concentration of NaOH (%) | Extraction time (hour(s)) | 1 | 2 | 3 | 4 | 5 |
| Haur Hejo      | 4 | 284 | 256 | 336 | 344 | 413 | 327 |
|                |              | 298 | 408 | 405 | 367 | 279 | 351 |
|                |              | 397 | 557 | 560 | 413 | 357 | 457 |
| Tali           | 4 | 529 | 560 | 474 | 549 | 552 | 533 |
|                |              | 512 | 577 | 558 | 667 | 357 | 534 |
|                |              | 592 | 595 | 562 | 519 | 519 | 535 |
| Gombong        | 4 | 441 | 343 | 493 | 470 | 341 | 418 |
|                |              | 120 | 211 | 208 | 104 | 255 | 180 |
|                |              | 172 | 57 | 79 | 58 | 62 | 86 |
| Haur Hejo      | 6 | 105 | 124 | 169 | 73 | 211 | 136 |
|                |              | 160 | 181 | 238 | 260 | 279 | 224 |
|                |              | 183 | 213 | 246 | 254 | 304 | 240 |
| Tali           | 6 | 614 | 507 | 541 | 499 | 621 | 556 |
|                |              | 840 | 700 | 624 | 743 | 642 | 710 |
|                |              | 758 | 592 | 640 | 598 | 652 | 648 |
| Gombong        | 6 | 222 | 216 | 201 | 81 | 175 | 179 |
|                |              | 120 | 211 | 208 | 104 | 255 | 180 |
|                |              | 43 | 79 | 81 | 76 | 54 | 67 |
The results of the single fiber tensile strength test of the three types of bamboo as shown in Table 2 are then processed in the form of histograms to facilitate the analysis of the effect of extraction time and NaOH concentration as shown in Figure 4. Based on Figure 4, it can be seen that the single fiber tensile strength average 543 MPa with a range of 357-667 MPa at 4% NaOH concentration and a 1-3 hour extraction process time, and the tensile strength of a single fiber bamboo Tali averaging 638 MPa with a range of 499-840 MPa at 4% NaOH concentration with a range extraction process time 1-3 hours. Meanwhile for bamboo Haur Hejo and Gombong at the time of extraction process 1-3 hours, the increase in NaOH concentration causes a decrease in the tensile strength of a single fiber adding concentration. The results of other researchers indicate that the tensile strength of a single bamboo fiber is 420 MPa, 383 - 862 MPa [4,28].

![Figure 4. Effect of varying NaOH concentration (%) and extraction time (hour) to tensile strength of single fiber.](image)

3.2. Content of hemicellulose, cellulose, and lignin
Data compilation results of cellulose and lignin testing on Tali and Haur Hejo bamboo for before and after the extraction process in NaOH solution are presented in table 3.

| Extraction process (in NaOH solution) | Chemical composition of bamboo fiber (%) |
|--------------------------------------|----------------------------------------|
|                                       | Hemicellulose | α-cellulose | Lignin   |
| 1. Bamboo Tali without extraction    | 17.29         | 46.91       | 21.30    |
| 2. Bamboo Haur Hejo without extraction | 19.15         | 52.3        | 20.01    |
| 3. Bamboo Tali with extraction       | 16.21         | 55.02       | 16.93    |
| 4. Bamboo Haur Hejo with extraction  | 16.38         | 55.68       | 18.28    |

![Table 3. Chemical composition of bamboo fiber.](image)
Based on table 3, it was seen that bamboo *Tali* had a reduction in lignin levels of 20.51% and an increase in $\alpha$-cellulose levels of 17.29%, while *Haur Hejo* bamboo had a reduction in lignin levels of 8.65% and an increase in $\alpha$-cellulose levels of 6.46%. The hemicellulose for both types of bamboo has a not significant decrease.

3.3. Effect cellulose content and SEM examination to tensile strength of single fiber

Table 1, which is clarified by figure 4 and table 2, it can be understood that the tensile strength of a single bamboo polymer fiber is inversely bound to the level of lignin. In other words, the smaller the level of lignin in fiber will increase its tensile strength. Based on the SEM examination as shown in figure 5, it can be seen that the morphology of the fiber surface with the extraction process is rougher. Fiber surface roughness is predicted to occur in lignin extraction, which will increase cellulose levels and adhesion to fiber-matrix composites.

![Figure 5](image)

*Figure 5. Morphology fiber (a) without process extraction; (b) with process extraction.*

3.4. FTIR test

The FTIR test results as shown in figure 6, showed that the wave number spectrum of *Tali*, *Haur Hejo* bamboo and *Gombong* bamboo between 3000 - 3500 cm$^{-1}$ showed a O-H bond to change due to the extraction process of lignocellulose in a solution of NaOH to cellulose and lignin. The hydroxil group reacts with NaOH which causes an increase in OH concentration. The wave number spectrum area between 2000-2800 shows changes in the stretching peak between bamboo without extraction processes and with the extraction process, which is indicated by the increase in cellulose levels in bamboo as a result of the extraction process. The FTIR spectrum shows a large peak range because of the vibration of the O-H stretch at 3700–3200 cm$^{-1}$, the peak of the C-H stretches around 2900 cm$^{-1}$ and the short peak of 1647.21 cm$^{-1}$ representing the stretch of OH and C-H from the cellulose group. The FTIR results are very in accordance with Li et al. [19] and Sedan et al. [24].

Lignin is indicated in a range of wave numbers between 1500-1600 cm$^{-1}$ with an aromatic C = C group. The peak point in this area turns out that the lignin extraction process is only reduced by 20.51% for *Tali* bamboo. Changes in the peak point in the source between 1000-1100 cm$^{-1}$ and a decrease in the stem point, the results of the extraction process in the area of 896 cm$^{-1}$ have an increase in the peak point in the bamboo extraction process, this shows that the extracellular process of stretching C-O and C-O-C bonds which means there is a stretch of the acetyl hemicellulose group and the formation of cellulose glycosides bonds.
Figure 6. Spectrum of functional groups resulting from the FTIR test.

The spectrum at the absorption peak is around 1500-1600 cm$^{-1}$ representing the lignin compound (aromatic C=C), the spectrum at absorption of 1735-1739 cm$^{-1}$ represents the carbonyl (C=O) stretching the acetyl hemicellulose group. The peak C-O stretch of about 1000-1100 cm$^{-1}$ from the cellulose group and the C-O-C peak around 896 cm$^{-1}$ stretching cellulose β-glycosidic bonds similar to Cai et al. [22].
4. Conclusion
The results of the characteristics of 3 types of bamboo showed that Tali, Haor Hejo bamboo had the potential to become natural fibers which replaced synthetic fibers. Diversification of bamboo natural fibers into composite products for various applications is directly able to increase bamboo's added value and at the same time provide solutions to environmental problems as a result of the application of synthetic fibers. The parameter of lignin extraction process from lignocellulose to produce cellulose with NaOH solution was influenced by the concentration of NaOH solution, extraction time and temperature and initial fiber diameter. The effectiveness of the lignin extraction process can be shown by the results of FTIR and fiber morphology using SEM.

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