THE EFFECT OF BAMBOO MICROSTRUCTURE ON THE MECHANICAL PROPERTIES OF BAMBOO FIBRE REINFORCED COMPOSITES.

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ABSTRACT

Three different bamboo species of two different ages are investigated in this research. The bamboos were sourced from South America, Europe and Southeastern Asia. Fibre tensile tests and impregnated fibre bundle tests were performed to characterise the mechanical properties. The results obtained from both tests were in agreement. Fibre stiffness values between 33-41 GPa were recorded and strengths between 331-509 MPa. The species grown in Europe showed lower stiffness for a mature plant, and lower strength for a plant younger than 1 year. Chemical analysis could not indicate a difference in composition in relation to the age of the plant. However the cellulose content seems to be related to the stiffness and strength obtained from the impregnated fibre bundle tests.

1 INTRODUCTION

Bamboo has a long history in traditional craftworks. In recent years, a shift is seen to use the plant for more technological applications like panels, plywood and also composite materials. The interest for composite applications is based on an interesting bio-economical mix; bamboo is fast-growing, has almost no need for fertilization, is abundantly present, has a high CO₂ sequestration capacity and in addition has favorable fibre mechanical properties.

Through the years the plant has drawn the attention of different research disciplines, particularly in the Plant Sciences. Botanical research might help us to understand the influence of the microstructure of bamboo on the composite properties. In biological research a bamboo fibre mostly refers to an elementary fibre, a cell. While in composite research, technical fibres (a bundle of elementary fibres) are embedded in a matrix to form a composite. Botanical research of Liese et al. [1] indicated different vascular bundle types amongst the different bamboo species. The influence on the ease of extraction and the obtained technical fibre mechanical properties is not yet known.

A large effort also went in determining the influence of age on the maturity of the plant. However, literature does not seem to agree on when the bamboo can be claimed mature. Some researchers state that full lignification of the elementary cell wall takes place within one growing season [2, 3]. Others find full lignification at 3 years and only 50 % lignification at 1 year [4] while yet other research by Lin et al. [5] shows the lignification process may take up to 7 years. However, little is still known about the influence of the lignification of the elementary bamboo fibres on the composite properties.

A question that needs to be asked is which bamboo species are adequate for composite applications, taking into consideration also the age of the plant. In addition there are several possibilities on how to extract technical bamboo fibres from the culm. All of these elements affect the properties of the final fibre. Therefore, there is a need to gain deeper understanding in the relationship
between the microstructure of the plant and the performance at composite level, with fibres of different plants extracted with the same technique.

2 MATERIALS AND METHODS

Three bamboo species were selected, sourced worldwide. The Guadua Angustifolia Kunth (GAK) from South America, the Dendrocalamus Membranaceus Munro (DMM) from Southeastern Asia and the Phyllostachys Nigra Boryana (PNB) grown in Europe. Of each species two ages were selected; age determination was done by visual features indicating the age range of the bamboo species. GAK was sourced of 1-3 years old and 3-5 years old, DMM of 1 year old and of 3 years old and PNB of 0.75 years and 2-4 years old. The fibres were extracted following a purely mechanical procedure, developed at KU Leuven. The starting material was dry and rewetted for 6-13 weeks prior to extracting the fibres.

In order to determine the mechanical properties of the extracted technical fibres, fibre tensile tests were performed according to the procedure described in previous work [6]. The methodology is based on ASTM C1557-14 but in addition strains are measured via optical strain measurement and digital image correlation to avoid phenomena such as slip and to obtain accurate stiffness results. The fibre density of each fibre type was determined with a gas pycnometer and used to estimate the cross section of the fibres for the tensile test. Tensile tests were performed on an Instron 5943 equipped with a 100 N load cell in a conditioned environment at 50% RH and 21°C. Technical fibres were visually inspected to avoid ones with major damage. The fibres were glued into a frame with a gauge length of 5 cm which was pneumatically gripped with a gripping force of 200 N. Two speckled optical flags were attached in order to measure the strain. A crosshead displacement rate of 1.5 mm/ min was applied. A minimum of 13 fibres was tested per species.

Impregnated fibre bundle (IFBT) tests were prepared [7]. Unidirectional composites were prepared and the stiffness and strength were back calculated from the composite properties via the rule of mixtures. IFBT gives a direct measurement of the average fibre properties. The bamboo fibres were dried for at least 4 days at 60°C prior to production and weighing. Epikote 828 LVEL epoxy with a 1,2-diaminocyclohexane (Dytek DCH-99) hardener from Hexion was used as matrix. Curing was done at 75°C for 1 hour followed by a subsequent postcuring of 1 hour at 150°C. Composites of 150 mm x 7.5 mm x 0.9 mm were made with a target volume fraction of 40 % fibres. Tensile tests were performed according to the ASTM D3039 standard on an Instron 4467 with a 5 kN loadcell and a crosshead displacement of 2 mm/min. The specimen gauge length was 7 cm and an extensometer with 2.5 cm gauge was used to measure the strain on the specimen. A minimum of 4 samples was tested per species. The Young’s modulus was calculated as the tangent of the stress-strain curve between 0.1-0.3 % strain.

Gas pycnometer measurements were performed to determine the density of the fibres, using a Quantachrome multipycnometer model MVP-D160-E with helium gas at a pressure of 1.5 bar as displacement medium. The fibres were dried prior to milling for 1 week in an oven at 60°C. By milling the fibres all the particles became smaller than 250 µm. The fibre dust was dried in a vacuum oven for at least 4 hours at 40°C before measuring.

To determine the chemical composition, i.e. the lignin, cellulose and hemicellulose content, an adapted Van Soest method in-house developed by Sweygers et al. (2017) was used. Fibres were prepared in the same way as for the gas pycnometer measurement. In the Van Soest method in the first step, a neutral detergent solvent (Na-lauryl sulphate, EDTA, pH = 7) is applied, thereby extracting the digestible cell contents (soluble sugars, starch, etc.) while the hemicellulose, cellulose and lignin fractions remain. In a second step, an acidic detergent solvent is applied (1 M H2SO4) to extract hemicellulose, while cellulose and lignin remain as a solid residue. In a third step concentrated sulfuric acid (72 w%) is applied to extract cellulose, whereby lignin remains as the last cell wall component. The measurements are performed in threefold, each measurement requiring 1 g of material.
3 RESULTS AND DISCUSSION

3.1 Microstructural characterisation

Based on the classification in four basic types of vascular bundles by Liese et al. [1] the considered species can be assigned as follows: Guadua Angustifolia Kunth and Phyllostachys Nigra Boryana consist out of vascular bundles of type I [8] and the Dendrocalamus Membranaceus Munro out of vascular bundles of type III, see Figure 1.

![Figure 1 Cross section of the considered bamboo species: Guadua Angustifolia Kunth (GAK); Dendrocalamus Membranaceus Munro (DMM); Phyllostachys Nigra Boryana (PNB).](image)

From the cross sectional images the areal percentage of fibre bundles (fibre sheaths + fibre strands) is calculated. For GAK and DMM the inner and outer 2 mm are excluded from the calculation and for the PNB, the inner and outer 1 mm. These regions are not economical to extract fibres from. For GAK the fibre bundles cover 20.8%, for PNB 21.9% and for DMM 30.5%. The calculations were performed on the samples of oldest age of the respective species. Figure 2 shows an image of the outlines of the fibre bundles used for the calculation. In the work of Londono et al. [9] the percentage of vascular bundle for the basal, middle and apical segment is given for Guadua Angustifolia Kunth. In the middle segment of the plant 26 % of the plant’s cross section is covered by vascular bundles. From that 26 %, 75% consists out of fiber bundles. This leads to comparable fibre bundle percentages as in this research.
3.2 Fibre tensile tests and impregnated fibre bundle tests

In order to estimate the fibre cross section, the density of the fibres was measured. Figure 3 shows that the fibre density of the different species does not vary a lot. Values between $1.38 \pm 0.01$ and $1.44 \pm 0.01$ could be found. This means that the difference between the different species/ages is less than 5%. Table 1 gives an overview of the measured fibre densities and the average calculated diameter of the fibres used for the fibre tensile test as well as the amount of fibres tested. The density of PNB 0.75 was not measured due to a shortage of material; for the calculations the same value as for PNB 2-4 is used. The finest extracted fibres are found for the GAK 3-5 and DMM 1 species.
Table 1 Overview of the test samples for the fibre tensile test and the IFBT.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>Specimens Fibre tensile test</th>
<th>Specimens IFBT</th>
<th>Fibre density (g/cm³)</th>
<th>Average diameter (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GAK 1-3</td>
<td>18</td>
<td>5</td>
<td>1.41 ± 0.03</td>
<td>185 ± 63</td>
</tr>
<tr>
<td>GAK 3-5</td>
<td>17</td>
<td>6</td>
<td>1.44 ± 0.01</td>
<td>144 ± 31</td>
</tr>
<tr>
<td>DMM 1</td>
<td>13</td>
<td>5</td>
<td>1.41 ± 0.02</td>
<td>134 ± 23</td>
</tr>
<tr>
<td>DMM 3</td>
<td>13</td>
<td>4</td>
<td>1.42 ± 0.02</td>
<td>228 ± 75</td>
</tr>
<tr>
<td>PNB 0.75</td>
<td>16</td>
<td>4</td>
<td>*1.38</td>
<td>180 ± 51</td>
</tr>
<tr>
<td>PNB 2-4</td>
<td>18</td>
<td>5</td>
<td>1.38 ± 0.01</td>
<td>167 ± 36</td>
</tr>
</tbody>
</table>

*was not measured, for the calculation estimated equal as PNB 2-4

Figure 4 Results Young’s modulus, tensile strength and failure strain of the fibre tensile test on the different bamboo species.

In Figure 4 the results of the fibre tensile tests are depicted. The lowest modulus of $33 ± 8$ GPa is recorded for GAK 1-3 and for the DMM 1 the highest value of $41 ± 7$ GPa was found. The highest recorded value is 25 % bigger than the lowest. The standard deviation on the Young’s modulus ranges from 17-28 %. The stiffness values are comparable to what is reported in literature [8, 10, 11].

For the tensile strength the lowest recorded value was $331 ± 111$ MPa for the DMM 3 species and the highest tensile strength was found to be $509 ± 149$ for the DMM 1. The
highest value is 50 % higher than the lowest. The standard deviation ranges between 27-36 %. For the tensile strain the lowest strain to failure is 0.9 ± 0.3 % for the PNB 0.75 and the highest 1.24 ± 0.42 % for the GAK 3-5. The largest value is 37% higher than the smallest. Mechanical extraction is known to give superior properties regarding stiffness and strength. However in this case, the strength values are rather low compared to literature [8, 10-12].

It can be stated that the spread on the strength and failure strain is higher than the spread on the modulus results. This is explained by the fact that strength and strain are defect sensitive whereas the stiffness is less sensitive to imperfections [7].

Figure 5 to Figure 7 compare the results obtained from the fibre tensile tests with the results back calculated from the IFBT. For most of the species the standard deviation is reduced in the IFBT test, for the stiffness, strength and strain. This is in agreement with literature [13], where it was shown that the standard deviations for natural fibre composites are comparable to those of glass fibre composites. In the IFBT more fibres are tested at once and properties are averaged out, both leading to a smaller deviation.

A two sided t-test ($\alpha = 0.05$) was performed to determine if there is a significant difference between the means obtained via the two tests. Only for the Young’s modulus of DMM 3, the difference is significant. For the tensile strength only the means of the PNB 2-4 species are significantly different and for the failure strain, a significant difference can be found for the DMM 1 species. Apart from this it can be concluded that the single fibre test gives comparable results as the IFBT in this research. In research on flax fibre composites, often higher strength values are obtained from the IFBT than from tests on technical fibres; in this case technical fibres suffer from defibrillation between elementary fibres and in the composite the elementary fibres can be bonded back together, leading to higher strength. This phenomenon is apparently absent in case of bamboo fibres.

Testing for the difference between the different species and ages, a few significant differences become clear. GAK 1-3 has a higher mean modulus value than all the other samples except for the DMM 1. No significant differences could be found between GAK 3-5, DMM 1 and DMM 3. Whereas PNB 2-4 has a significantly lower mean modulus than all the other samples except for PNB 0.75, compared to which no difference could be found.

Considering the tensile strength of the different samples, other results are found. There is no significant difference between GAK 1-3, GAK 3-5, DMM 1 and DMM 3. However, PNB 0.75 has a significantly lower strength than all the other samples. This may indicate that the material was too little matured.
Figure 5 Comparison of results of the Young's modulus from the fibre tensile test and the backcalculated Young's modulus from the impregnated fibre bundle test (IFBT).

Figure 6 Comparison results of the fibre tensile strength from the fibre tensile test and the backcalculated fibre tensile strength from the impregnated fibre bundle test (IFBT).
3.3 Chemical characterization

In literature data can be found for the Klason lignin content of the bulk bamboo material of different ages. Li et al. [14] saw a difference between 1 year old bamboo and 3 or 5 year old bamboo. The lignin content increased from 22.1 % to 23.5%. In the work of Itoh et al. [2] bamboo of one month old had a lignin content of 15.2 % which increased to 27.2 % for 3 months old bamboo. Further evaluation of bamboo of 2 to 14 years old did not reveal any change in lignin content, the average lignin content for mature bamboo at the 20th internode was 24.8 %.

Table 2 lists the results of the chemical characterization of the different species. A two sided t-test revealed that there is no significant difference in lignin content between the two considered ages for GAK and DMM. Taking into account the proximity to the lignin content values from literature and the fact that no difference could be found between the two ages, it can be stated that both ages of both species can be considered mature. However, significant differences in the cellulose content between GAK 1-3 and GAK 3-5 and DMM 1 and DMM 3 are revealed.

It is commonly accepted that cellulose is the main contributor to the stiffness and strength of the fibres. To investigate the effect we look at the IFBT results, which give an average value of the mechanical properties of the fibres. Looking at the modulus values of the IFBT, it can be seen that the samples with the highest cellulose content, DMM 1 and GAK 1-3 have high Young’s modulus. DMM 1 and GAK 1-3 are not significantly different and GAK 1-3 is significantly higher than the other samples.

Whereas looking at the ultimate tensile strength of the IFBT, fibres with low cellulose content seem to have lower strength properties at least for the PNB; this does not completely hold for the DMM 3. It is noticed that the fibres with low tensile strength also contain a higher amount of extractives. To determine the exact role of these extractives on the strength of the fibres, further investigation is needed.
Table 2 Results of the chemical characterization of the considered species.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>Cellulose (%)</th>
<th>Hemicellulose (%)</th>
<th>Lignin (%)</th>
<th>Ash (%)</th>
<th>Extractives (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GAK 1-3</td>
<td>53.97 ± 1.18</td>
<td>13.82 ± 0.31</td>
<td>30.97 ± 0.96</td>
<td>0.55 ± 0.05</td>
<td>0.70 ± 0.45</td>
</tr>
<tr>
<td>GAK 3-5</td>
<td>50.80 ± 0.23</td>
<td>14.39 ± 0.48</td>
<td>29.32 ± 0.06</td>
<td>4.99 ± 0.29</td>
<td>0.49 ± 0.42</td>
</tr>
<tr>
<td>DMM 1</td>
<td>58.35 ± 0.39</td>
<td>15.21 ± 0.27</td>
<td>26.65 ± 0.38</td>
<td>0.29 ± 0.19</td>
<td>~ 0</td>
</tr>
<tr>
<td>DMM 3</td>
<td>45.51 ± 0.24</td>
<td>15.15 ± 0.10</td>
<td>29.57 ± 0.66</td>
<td>0.11 ± 0.03</td>
<td>9.66 ± 0.63</td>
</tr>
<tr>
<td>PNB 0.75*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PNB 2-4</td>
<td>46.66 ± 0.16</td>
<td>15.67 ± 0.07</td>
<td>32.43 ± 0.38</td>
<td>0.50 ± 0.09</td>
<td>4.74 ± 0.41</td>
</tr>
</tbody>
</table>

* The chemical composition of PNB 0.75 could not be measured due to a shortage of material.

4 CONCLUSIONS

Three different bamboo species, each of two different ages were investigated. From the microscopical images it could be seen that the Dendrocalamus membranaceus Munro has a 50% higher percentage of fibre bundles present than the Guadua angustifolia Kunth and the Phyllostachys nigra Boryana considered.

The density of the fibres was determined and only differences less than 5% could be found between the different species and ages.

To investigate the mechanical properties, fibre tensile tests and impregnated fibre bundle tests (IFBT) were performed. The results of both tests were in agreement. It could be seen that the strength and failure strain properties showed higher variance than the modulus. This is explained by the fact that the stiffness is less defect sensitive. Also the IFBT showed smaller variance than the single fibre test, since more fibres are tested at once and the effects are averaged. GAK 1-3 showed an overall better stiffness, although not significantly different from DMM 1. And the PNB species performed less, PNB 2-4 had the lowest stiffness and PNB 0.75 the lowest strength.

Chemical analysis was performed to investigate the effect of age on the lignification and the relationship with the mechanical properties. No significant differences could be found between the lignin content of the two considered ages of two species; therefore it is concluded that both ages can be considered mature in this research.

The cellulose content seems to be related to the stiffness and even more pronounced to the strength of the fibres. What the influence of extractives could be on the strength of the fibres still asks for more investigation.

REFERENCES


