Citation	Delphine E.C. Depuydt, Nick Sweygers, Lise Appels, Jan Ivens, Aart W. van Vuure (2019) Bamboo fibres sourced from 3 global locations, a microstructural, mechanical and chemical composition study. Journal of Reinforced Plastics and Composites, 38 (9), 397-412.
Archived version	Author manuscript: the content is identical to the content of the published paper, but without the final typesetting by the publisher
Published version	https://doi.org/10.1177/0731684419828532
Journal homepage	https://journals.sagepub.com/home/jrp
Author contact	your email aartwillem.vanvuure@kuleuven.be your phone number <u>+32 16 30 11 38</u>

(article begins on next page)

Bamboo fibres sourced from 3 global locations, a microstructural, mechanical and chemical composition study.

Delphine E.C. Depuydt^{a*}, Nick Sweygers^b, Lise Appels^b, Jan Ivens^c, Aart W. van Vuure^a

^aKU Leuven, dept. Materials Engineering; Campus Groep T, Composite Materials Group, Andreas Vesaliusstraat 13,
3000 Leuven, Belgium

^bKU Leuven, dept. Chemical Engineering; Process and Environmental Technology Lab, Jan Pieter de Nayerlaan 5, 2860
Sint-Katelijne-Waver, Belgium

^cKU Leuven, dept. Materials Engineering; Technology Campus De Nayer, Composite Materials Group, Jan De
Nayerlaan 5, 2860 Sint-Katelijne-Waver, Belgium

*corresponding author, delphine.depuydt@kuleuven.be

Abstract

More than 1200 bamboo species are known, therefore the question arises which species are suited for composites and which microstructural characteristics of the plant are decisive. Three different bamboo species are considered, each of two ages. *Technical* fibre tensile tests and impregnated fibre bundle tests (IFBT) were performed to characterise the fibres and maceration was applied to determine the length over diameter (l/d) ratio of the *elementary* fibres. From the IFBT it is concluded that the stiffness of bamboo fibres from tropical species considered in this research is unaffected by the species nor the age of the plant. The strength is more affected by the region of growth, with the temperate bamboo species having lower strength as a result of a lower l/d ratio of the *elementary* fibre. Chemical analysis helped to investigate the age difference and it can be stated that the fibres are mature within one growing season.

Keywords: A. Fibres B. Mechanical properties D. Microstructural analysis D. Chemical analysis

1 Introduction

In light of the growing awareness of environmental problems, especially the use of petroleum-based products, natural fibre composites are emerging as a viable alternative to synthetic fibres. The specific mechanical properties of natural fibre composites can rival those of traditional materials, especially glass fibre composites [1].

The natural fibres can be divided into three categories: plant, animal and mineral fibres [2, 3], of which the first two can be considered as renewable. The plant fibres are composed out of: straw (wheat, rice), grass (bamboo, bagasse), bast (flax, hemp, jute), fruit (coir, kapok), leaf (abaca, banana, sisal), seed (cotton) and wood fibres [4, 5]. Bamboo belongs to the grass family Poaceae, where the tribes of the woody bamboos, Bambuseae and Arundinarieae, and more precise the ‘giant bamboos’ (that can grow to 30 m with a diameter of up to 30 cm [6]) are of special interest for composite applications [7]. Bamboo genera that have been identified as potential source for fibre extraction due to their anatomical characteristics and their common use in traditional construction are: *Bambusa*, *Dendrocalamus*, *Gigantochloa*, *Guadua* and *Phyllostachys* [8, 9].

Bamboo has a few advantages over other natural fibres, beside the fact that it grows fast, up to 21 cm a day, it is abundant [10] and can help with the reforestation of degraded land [11]. It is estimated that 15 million hectare of ‘giant bamboo’ is present [8], which can be used for fibre extraction. It is a very diverse plant, since over 1600 species are known [12] and spread worldwide. Bamboo is native to all continents except for Antarctica and Europe. The natural distribution of the bamboo can mostly be found in the tropical and sub-tropical climate regions [7], but some bamboo is native to temperate climate zones and can therefore adapt to grow in European regions [13]. INBAR compared the CO₂ sequestration of Ma bamboo (*Dendrocalamus latiflorus*) and an Eucalyptus plantation and found that they have comparable carbon sequestration capacity as long as the bamboo forest is managed and that for both species valuable products are made that fixate the carbon. The accumulated carbon content after 10 years is 128 t C/ha for Ma bamboo and 115 t C/ha for Eucalyptus [14].

There are different possibilities to extract bamboo fibres as explained by Zakikhani et al. [15]. KU Leuven has developed a purely mechanical extraction technique which results in the extraction of long, largely undamaged fibres [8, 16]. Up till now, this method was mainly applied to extract fibres from the *Guadua angustifolia* Kunth [1, 17, 18] but the question raised how well fibres of other bamboo species are suited for composite applications. For flax and hemp fibres, a variation in mechanical properties is found depending on the species. For flax, Young’s moduli between 49-68 GPa are reported and for hemp between 20-45 GPa [19, 20].

Figure 1 gives an exploded hierarchical view from culm to microfibril, demonstrating the multicomponent structure of a bamboo plant. The plant is optimized in order to withstand bending induced by external factors like wind, with a minimum of material. The hollow culm is reinforced by nodes, where the nodal diaphragm gives extra stiffness to the culm and helps to prevent buckling [21]. The nodes appear with a certain regularity along the height of the culm. In the cross section of the culm, vascular bundles, containing the bamboo fibres, can be distinguished. The vascular bundles are longitudinally aligned in the culm and surrounded by a matrix of parenchyma tissue. In a vascular bundle the phloem, protoxylem and metaxylem, having the function of conducting tissue are present, as shown in the detail of Figure 1. Generally the metaxylem vessels are attached by four fibre sheaths (sclerenchyma sheaths), though some species have additionally isolated fibre strands that carry out the function of mechanical support. The concentration of the vascular bundles increases from the inner part to the outer part of the culm, making bamboo a functionally graded material, perfectly adapted to high specific bending stiffness and strength [22, 23].

Different types of vascular bundle were distinguished by Grosser & Liese [24]. Figure 2 gives an overview of the 4 most common types. Differences can be found in the presence of additional isolated fibre strands. Both the fibre sheaths and the strands are referred to as *technical* fibres once extracted from the plant. Their length is equal to the internodal length, since in the node, the longitudinal orientation of the fibres is lost as they get entangled. Typical length and diameter of the *technical* fibres is 20-35 cm and 90-280 μm respectively [8].

Each *technical* fibre (or fibre bundle) consists out of a smaller building unit, named *elementary* fibres (a single plant cell). On a cross sectional view they are recognized by their hexagonal and pentagonal shapes, with a small hole, the lumen in the middle [15]. In between the *elementary* fibres, the middle lamellae are found, the ‘intercellular glue’ that binds the cells together. The typical diameter and length for *elementary* fibres ranges from 10-40 μm and 1.0-4.3 mm respectively with lumen sizes in between 2-20 μm [12, 25]. To separate the *technical* fibre into *elementary* fibres, tweezers or chemical techniques, referred to as maceration, can be used [26]. Via chemical techniques the middle lamellae are broken down and the fibres get separated. This technique has already extensively been applied to wood, but has proven to work for other natural fibres as well [27, 28].

An *elementary* fibre is made out of separate layers, with the bulk (80%) consisting out of the secondary wall, where highly crystalline cellulose microfibrils are embedded in a hemicellulose and lignin matrix. The model of Parameswaran and Liese [29] depicts the secondary wall of the *elementary* bamboo fibres as a polylamellate structure made out of a number of alternate thin and thick layers. In each layer the microfibrils are oriented with a certain angle, referred to as the microfibril

angle (MFA). In the thick layers, the angle is close to 2-5° while in the thin layers it is close to 85-90° [29]. It is known that the MFA is inversely correlated with the mechanical properties of plant fibres [30]. Yu et al. [31] determined the MFA of 11 bamboo species and showed that the MFA variation between species is small, with microfibril angles ranging from 7.5° to 10.6°.

As a bamboo plant grows, tissue will develop in 4 phases: cell differentiation, cell elongation, cell wall thickening and lignification [26, 32]. Gritsch et al. [32] investigated the development of *elementary* bamboo fibres and parenchyma of the *Dendrocalamus asper* and was able to point out that lignification can take place prior to cessation of elongation of the fibres. The process of lignification is also referred to as maturation and is described as “the formation of a polymer within the cell wall that provides strength to the culm” by Liese et al. [12]. The general consensus is that a bamboo culm matures at about 2-3 years and then reaches its maximum strength [12], however literature does not seem to agree on when the lignification process stops.

Itoh et al. [33] studied the Klason lignin content of *Phyllostachys heterocycla* bamboo fibres and parenchyma and found that after 7 months the maximum lignin content was reached. After that the lignin content stayed fairly constant in the mature culm, between 23.2 and 26.4%, from 2 to 14 year. Lybeer et al. [21] came to the same conclusion that full lignification of the cell wall is completed within one growing season by analysing the diameter, wall thickness and amount of wall layers of the *elementary* fibre of the *Phyllostachys viridiglaucescens*. For plants older than one year no significant increase in wall thickness of the fibres and parenchyma cells could be found. Murphy and Alvin [34] studied the same characteristics as Lybeer et al. [21] on *elementary* fibres of the *Gigantochloa scortechninii* Gamble and they found that between 2 and 3 years there was still an increase in wall thickness, and it is changing from the inside to the outside of the culm. They also stated that the late maturing fibres in the fibre strand have a larger diameter and therefore the potential to deposit extra layers longer. The heterogeneity in the fibre development was also noticed by Gritsch et al. [35] where it was shown that *elementary* fibres close to the phloem mature at a faster rate. Additionally, they showed that the number of wall layers is independent of the wall thickness of the cell and the number of wall layers can be the same of *elementary* fibres at the periphery or close to the phloem. Li et al. [36] analysed bamboo culms of the *Phyllostachys pubescens*, focussing on the Klason lignin content. It was found that 1 year old bamboo had a lower Klason lignin content than 3 and 5 year old culms, which were similar. However, a 36% increase in bamboo density was found from one to three year old bamboo, and only a slight increase of 3% from three to five years. This increase is related with the deposition of additional wall layers, since the vascular bundles per unit area do not change with age. Lin et al. [37] studied the extent of lignification via visible light absorption microscopy of the *Phyllostachys pubescens* Morel fibres and parenchyma and found that the

lignification takes up to 7 years. In this research it is investigated how the age and different degree in lignification of the fibres affects the mechanical properties.

An overview of the mechanical properties found in literature of *technical* bamboo fibres is given in Table 1 of Depuydt and Hendrickx et al. [38] Young's moduli vary between 19-43 GPa and strength between 341-860 MPa. Different factors can be responsible for the large variation. As mentioned before, different extraction methods, ages of the plant but also the used test method can lead to variation in the measured properties. In this research the link between the microstructure of different bamboo species is investigated, using the same extraction method, and *technical* fibre tensile test method as developed in previous research [38]. In this way the authors try to reveal an unbiased view of the influence of age and species on the mechanical properties. Via this way deeper insight in the suitability of bamboo species for composites is sought.

2 Materials and Methods

2.1 Material and extraction

Three bamboo species were selected, sourced worldwide. The *Guadua angustifolia* Kunth (GAK) sourced from South America, the *Dendrocalamus membranaceus* Munro (DMM) from Asia and the *Phyllostachys nigra* Boryana (PNB) from Europe. GAK is a tropical species, that was harvested from the Coffee Region, at 1300 meters above sea level with an annual average temperature of 23 °C and annual average precipitation of 2200 mm and relative humidity of 80% [8]. The GAK is the most economically important bamboo in the Western Hemisphere [7]. The DMM was harvested from the Hoa Binh province, 60 km west from Hanoi having a tropical climate, with an average rainfall of around 1800 mm, an average temperature of 24°C and a relative humidity of 80% [39]. The PNB was harvested in the province of Limburg in Belgium, 45 m above sea level with an annual average temperature of 10°C and an annual average precipitation of 850 mm and relative humidity of 78% [40]. The habitat of *Phyllostachys* species is a warm-temperate climate. They are also known to be winter hard, which makes them suitable to grow in Europe as well [7]. Figure 3 gives an overview of the natural distribution of bamboo and different genera used in this study and the places of harvest.

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 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 [18]. The starting material was dry and rewetted for 6-13 weeks prior to extracting the fibres.

2.2 Microscopy and areal fibre bundle fraction determination

Radial sections of 1 cm in width were cut out of each bamboo species and embedded in an EpoxiCure 2 Epoxy mounting resin and EpoxiCure 2 hardener from Buehler. The samples were sanded and polished and analysed with a light microscope (Axioskop 40, Zeiss) to study the areal fibre bundle fraction. The microscopic images were processed with image J by applying a threshold filter which separated the fibre bundles from the surrounding parenchyma. By dividing the area occupied by fibre bundles by the total culm cross section, the areal fibre bundle fraction was determined. This value represents the maximum achievable fibre extraction percentage.

2.3 Diameter determination of extracted fibres

The diameter of the extracted fibres was determined in order to study possible differences in extraction of the fibres. The algorithm for the diameter determination was explained by Hendrickx et al. [41] and starts from a flatbed scanned image at 1200 dpi. After putting a threshold to separate the fibres from the background and morphological operations to clean the image, the fibre diameter was determined by taking the local maxima of the Euclidean distance transform function for every pixel along the fibre length. Around 30 *technical* fibres were measured for each species with a length of around 15 cm, leading to more than 200.000 diameter measured points.

2.4 Gas pycnometer

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 with a centrifugal mill, type Retsch ZM100 (Gemini BV, Netherlands), all the particles became smaller than 250 µm. The fibre dust was dried in a vacuum oven for at least 5 hours at 30°C before measuring. The measured density can be considered as the cell wall density of the bamboo fibre.

2.5 Fibre tensile test

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 [38]. The methodology is based on the ASTM C1557-14 standard

but in addition the strain was 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 is determined with a gas pycnometer and used to estimate the cross section of the fibres for the tensile test. *Technical* fibres were randomly selected but visually inspected to verify absence of major damage. The fibres were preconditioned at 50% relative humidity and 21°C for at least 24h. Tensile tests were performed in the same conditioned environment on an Instron 5943 equipped with a 100 N load cell. 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 optical speckled 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 were tested per species.

2.6 Impregnated fibre bundle test

Impregnated fibre bundle (IFBT) tests were prepared conform the work of Bensadoun et al. [42]. Unidirectional composites were made and the stiffness and strength were back calculated from the composite properties via the rule of mixtures, using Eq.1 and Eq. 2. With E_f and E_m the fibre and matrix modulus, σ_f and σ'_m the fibre and matrix tensile stress (at the fibre failure strain) and V_f and V_m the fibre and matrix volume fraction. IFBT gives a direct measurement of the achievable fibre reinforcement within the composite. It has been pointed out by Shah et al. [43] that the properties of bast fibres differ when measured on elementary fibre level or technical fibre level or are back calculated from the composites properties. This research performs both measurements on a *technical* fibre level as on composites, in order to gain insight in the behaviour of bamboo fibres.

$$E_c = V_f E_f + V_m E_m \quad (1)$$

$$\sigma_c = V_f \sigma_f + V_m \sigma'_m \quad (2)$$

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 post curing of 1 hour at 150°C. Composites of 150 x 7.5 x 0.9 mm were made with a target volume fraction of 40 % fibres, which was calculated based on the weight and measured density of the fibres. Tensile tests were performed according to the ASTM D3039 standard on an Instron 4467 with a 5 kN load cell and a crosshead displacement of 2 mm/min. The specimens were gripped with pneumatic grips of 6 bars. The specimen gauge length was 7 cm and an extensometer of 2.5 cm was used to measure the strain of the specimens. A minimum of 4 samples was tested per species. The Young's modulus was calculated as the tangent at the stress-strain curve between 0.1-0.3% strain. To detect significant differences between group means, a two-sided t-tests was performed with a significance level of 0.05.

2.7 Maceration

From the three considered species, the youngest age was selected to study the length over diameter ratio of the *elementary* fibres. Random selected pieces of around 5 cm in length of *technical* fibres were immersed in the Franklin maceration solution [44] to split the *technical* fibres into *elementary* fibres. The fibres were immersed in a mixture of hydrogen peroxide (35%) and concentrated acetic acid (1:1 v/v) and kept at a temperature of 60°C for 24h while stirring. After the procedure the fibres were filtered under vacuum using a polycarbonate filter (Whatman Nuclepore) of 2 µm mesh size and subsequently washed 3 times with distilled water. After that, the fibres were stained with Safranin O and Astra blue as described in Coz et al. 2002 [45]. The fibres were subsequently dried in an oven at 60°C. Glass slides were prepared by placing a droplet of glycerine on the glass slide and arranging the *elementary* fibres on top. The fibres were covered with a cover slit, which was sealed with nail polish. The glass slides were scanned at a resolution of 6400 dpi (1 pixel = 4 µm) with a scanner (Epson perfection V550 photo) in order to determine the length of 50 fibres per species. This was done manually via image J software. The diameter of the fibres was measured at larger magnification with a light microscope (Axioskop 40, Zeiss) at two distinct points.

2.8 Chemical characterization

To determine the chemical composition, i.e. the lignin, cellulose and hemicellulose content, an adapted Van Soest method developed by Sweygers et al. [46] was used. Fibres were milled to powder in the same way as described for the gas pycnometer measurements. In a first step, a neutral detergent at 100°C was applied, thereby extracting the digestible cell contents (soluble sugars, starch) while the hemicellulose, cellulose and lignin fractions remain. In a second step, the residual content was treated with sodium chlorite and acetic acid at 75°C in a water bath, the loss in matter is attributed to the lignin fraction. The remaining fraction, being the holocellulose, is treated with a mildly acidified detergent at 100°C, which only leaves cellulose as the sole particulate matter. The ash content is determined by heating the fibre particles for 2h at 550°C in a muffle furnace. The residual inorganic matter is the ash content. The cellulose, hemicellulose and lignin percentages mentioned in this report were determined by subtracting the ash content and extractives from the batch, and rescaling the former components to 100%. The measurements were 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. [12] the considered species can be assigned as follows: *Guadua angustifolia* Kunth and *Phyllostachys nigra* Boryana consist out of vascular bundles of type I [18, 24] and the *Dendrocalamus membranaceus* Munro out of vascular bundles of type III, see Figure 4. Figure 4 shows the cross section of the three different bamboo species for both ages considered. From the cross sections, the difference in culm wall thickness can be derived, with the *Guadua* showing the thickest wall of around 12 mm and the *Phyllostachys* grown in Europe the smallest wall thickness of 3.5 mm.

Figure 5 indicates how the areal percentage of fibre bundles (fibre sheaths + fibre strands) was calculated. From the pictures of the DMM 1 it is seen that the fibre strand possesses lower contrast with the matrix. This is because the fibres are less mature, having larger lumen and resembling more the open structure of the parenchyma [34]. When the bamboo matures, these are the last regions to develop.

The areal fibre bundle fraction (%) of the *Guadua* and *Phyllostachys* are comparable and between 23-24% for both ages. The *Dendrocalamus* has a higher fibre bundle density of 26% for the DMM3 and 29% for the DMM1; therefore 10-25 % higher than for the other species. The variation between the two ages is a consequence of natural variation, since the samples are not taken from the same culm. When a bamboo plant ages, no additional fibre bundles are created, only additional cell walls are deposited. Nevertheless, the *Dendrocalamus* shows a higher fibre bundle fraction, which leads to a higher potential for fibre extraction. When studying the pictures, it seems that this might be related with the vascular bundle type, since the additional large fibre strand, makes the fibres stand closer to each other.

In the work of Londono et al. [10] the percentage of vascular bundle for the basal, middle segment and apical segment is given for the *Guadua angustifolia* Kunth. In the middle segment of the plant 26% of the plants cross section is covered by vascular bundles. Within the vascular bundle, the fibre percentage fluctuates between 64.8 % and 97.2 % along the culm thickness. Assuming an average fibre percentage of 80%, this leads to comparable fibre bundle percentages as in this research.

Figure 6 gives an overview of the diameter distribution of the extracted fibres, diameters are recorded ranging from 50 µm to more than 1600 µm. In literature smaller diameters are reported for the *Guadua angustifolia* Kunth by Trujillo et al. [17] ranging between 90-280 µm. Considering the cross sections of the bamboo species and the dimensions of the bamboo fibre

sheaths from Figure 4, it can be concluded that extracting fibres from rewetted bamboo culms is less efficient and leads to agglomerates of *technical* fibres. During the first desorption cycle, irreversible hydrogen bonds are created, which prevent the bamboo to take its original shape when rewetting and in this way making fibre extraction more difficult. This idea is not new, a similar hypothesis is found in the study on the hysteresis of natural fibres and wood [4, 47].

For the GAK and PNB a broad distribution is seen, whereas the DMM shows a peak at smaller diameter, between 100-150 μm , for both ages. This peak can be explained looking at the extracted fibres as shown in Figure 7. The DMM fibres demonstrate a more fibrillated look, being more divided into split fibre sheaths after extraction as compared to the GAK and PNB.

3.2 Tensile tests

3.2.1 Fibre tensile tests

To determine the fibre cross section of the tensile tested fibres, a gravimetric approach was used, therefore the cell wall density of the fibres was measured via a gas pycnometer, by first milling the fibres. The fibre density of the different species does not vary a lot; values between $1.38 \pm 0.01 \text{ g/cm}^3$ and $1.44 \pm 0.01 \text{ g/cm}^3$ were found. This is in agreement with literature, where Osorio et al. [18] determined the density of dry freshly extracted GAK fibres to be 1.4 g/cm^3 . In literature the density of the complete bamboo culm can be found, and ranges between $0.4\text{-}0.9 \text{ g/cm}^3$, which is as would be expected lower, due to the porosities that are present when considering the whole system [12, 22]. In this research it is found that the difference in fibre density between the different species and ages is less than 5 %. Table 1 gives an overview of the measured fibre densities and the average calculated diameter of the fibre used for the fibre tensile test as well as the amount of fibres tested. For the *technical* fibre tensile test, fine fibres were selected, to preferably test split fibre sheaths. The finest extracted fibres are found for the DMM 1 and GAK 3-5 species.

In Figure 8 the results of the *technical* fibre tensile tests are depicted, as the white bars. All species and ages showed linear behaviour up to failure. The lowest Young's modulus of $33 \pm 8 \text{ GPa}$ is recorded for GAK 1-3 and for the DMM 1 the highest Young's modulus of $41 \pm 7 \text{ GPa}$ was found. The highest recorded value is 25 % larger 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 [18, 48, 49], though lower than previous own work [38]. For bamboo fibres extracted from a fresh *Guadua* plant [38], a Young's modulus of $52 \pm 7 \text{ GPa}$, a tensile strength of $658 \pm 135 \text{ MPa}$ and a strain to failure of $1.33 \pm 0.2 \%$ were found. Extracting from a rewetted plant leads to thicker fibre bundles, with bigger agglomerates containing parenchyma

between the fibres. The presence of parenchyma and possibly more induced defects during extraction, can be pointed out as the reasons for the lower measured properties.

For the fibre tensile strength the lowest recorded value is 313 ± 101 MPa for the PNB 0.75 species and the highest tensile strength was found to be 509 ± 149 MPa for the DMM 1. The highest value is 60 % higher than the lowest. The standard deviation ranges from 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 %. The highest 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 [18, 48-50].

It can be stated that the spread on the strength and strain to failure is higher than the spread on the modulus results. This is explained by the fact that strength and strain to failure are defect sensitive whereas the stiffness is less sensitive to imperfections [42]. The fact that the strength is defect sensitive and in this case lower than the strength recorded for bamboo fibres extracted from a fresh culm, supports the hypothesis that more damage is initiated when extracting from a rewetted culm.

Previous research by Trujillo et al. [17] performed on bamboo fibres extracted from a fresh *Guadua angustifolia* Kunth plant have shown that a length-based model holds for the strength prediction of bamboo fibres. This means that the failure probability was determined to be independent of the diameter of the fibre. Although less fibres were tested in this research, a plot has been made to check the correlation between the fibre diameter and the fibre strength, as shown in Figure 9a. Low R-square values between 0 and 0.24 are found for the different species and ages. Therefore there is no reason to believe the strength results would be biased by diameter differences. The relation between the Young's modulus and the fibre diameter was also assessed in Figure 9b, and no correlation could be found either, indicated by the low R-square values ranging from 0.02 to 0.16. This means that the stiffness results for larger fibres are representative, even though they might contain more parenchyma.

3.2.2 Impregnated fibre bundle tests

In Figure 10 a typical stress strain curve of the composites made with the different species and ages is given and a linear behaviour is detected as well which is in contrast with the non-linear behaviour noted for flax fibres composites [51, 52]. In Figure 8 the orange bars show the results of the back calculated properties from the impregnated fibre bundle tests and Table 2 summarises the results. For most of the species the standard deviation is reduced, both for the Young's modulus, strength and strain to failure. This is in agreement with literature, where it was shown that the standard deviations for

natural fibre composites are comparable to those of glass fibre composites [53]. In the IFBT more fibres are tested at once and properties are averaged out, both leading to a smaller deviation. Since more fibres are tested in the IFBT than in the fibre tensile test, these results will be further used to check if there is a correlation with the chemical composition of the species.

The IFBT values are comparable with what was reported in the work of Perremans et al. [1] for unidirectional bamboo composites, a Young's modulus (E) of 17.6 ± 1 GPa, a strength (σ) of 222 ± 13 MPa and a strain to failure of 1.4 ± 0.2 % were measured (normalised to a fibre volume fraction of 40%). Via the rule of mixture the back calculated properties can be determined, taking into account the matrix properties ($E = 2.7$ GPa, $\sigma = 70$ MPa). This leads to a back calculated fibre modulus of 40 GPa and *technical* fibre strength of 498 MPa.

A two sided t-test ($\alpha = 0.05$) was performed to determine if there is a significant difference between the means obtained via the fibre tensile test and IFBT. For the Young's modulus of GAK 1-3 and DMM 3, the difference is significant. For the tensile strength none of the means are significant different and for the strain to failure, a significant difference can be found for the DMM 1 species. This means that most of the differences between the fibre tensile test results and the IFBT were not significant and therefore it can be concluded that the single fibre test gives comparable results as the back calculated properties from the IFBT in this research.

This is in contrast with the behaviour found for other natural fibres, including flax and sisal [38, 43, 54, 55]. For these fibres the properties measured on *technical* fibres tend to be lower than the back calculated properties from the composite. Therefore, as pointed out in the work of Shah et al. [43] "it is debatable whether the reinforcing unit within a composite is a *technical* fibre or *elementary* fibre". The difference between *technical* fibre tensile test and back calculated properties is found in the fact that when testing a *technical* fibre with a gauge length longer than the *elementary* fibre length, shear develops in the often pectin-rich middle lamellae and failure occurs at lower properties. The fact that this behaviour is absent for bamboo fibres can be explained by the structure of the fibre. Bamboo does not have pectin-rich middle lamellae, but instead lignin-rich zones, where the 3D polymer fixes the structure in a rigid way.

Testing for the difference in back calculated Young's modulus between the different species and ages based on the IFBT, a few significant differences become clear. GAK 1-3 has a higher mean value than all the other samples except for the DMM 1. No significant difference could be found between GAK 3-5, DMM 1 and DMM 3. Whereas PNB 2-4 has a significantly lower mean than all the other samples except for PNB 0.75, where no difference could be found.

Considering the back calculated 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. Additionally to that, PNB 0.75 and PNB 2-4 have a significantly lower strength than all the other samples. To further investigate the differences, the characteristics of the *elementary* fibres and chemical composition are analysed.

In general it seems that the stiffness and strength of species grown in tropical climates is not affected by the species nor the age of the plant. Comparing with the *Phyllostachys nigra* Boryana that was grown in Europe (temperate climate), it seems that the Young's modulus is only slightly affected (<15 % lower), but the strength is more affected (around 30% lower).

3.3 Influence of *elementary* fibre dimensions

Maceration of the *technical* fibres was performed in order to study the length over diameter ratio (l/d) of the *elementary* fibres. Figure 11 a)-c) shows the glass slides that were scanned from which the length of the *elementary* fibres was measured. The diameter was determined as shown in Figure 11 d) by measuring at two points the diameter with a light microscope. Figure 12 gives a plot of the 50 measured *elementary* fibres for each species, only the youngest age was analysed. It can be seen that the l/d ratio of the PNB 0.75 is smaller than for the other two species. For PNB 0.75 an average l/d of 87 ± 30 was calculated and 133 ± 45 and 178 ± 68 for GAK 1-3 and DMM 1 respectively. The average *elementary* fibre lengths of the three measured species are 1.3 mm, 2.8 mm and 3.1 mm for the PNB 0.75, GAK 1-3 and DMM 1 respectively. Liese et al. [12] mentions the average *elementary* fibre length of different bamboo species. For the *Dendrocalamus membranaceus* a length of 4.3 mm is stated. In previous work of the same author [25] a fibre length of 1.04 mm is mentioned for the *Phyllostachys nigra*, while for other *Phyllostachys* species lengths between 1.3 and 2.5 mm are stated [12]. The average *elementary* fibre length for the *Guadua angustifolia* Kunth is reported to be 2.1 mm [56]. This may lead to the conclusion that the *Phyllostachys* has a shorter l/d which could be responsible in this case for the lower measured mechanical properties. When considering the bamboo *technical* fibre as a composite on its own [56], where the short fibre composite micromechanics rules apply to, it can be understood that the strength is more affected by small l/d ratios in the order of 100 than the stiffness; the E-modulus typically reaches the level of a continuous fibre composite above l/d ratios of 50 [reference Jim Thomason]. This can explain why a bigger difference is found in the strength of the fibres, than the stiffness comparing the different species.

To further investigate the question whether the region of growth has an effect on the properties, the culm properties are compared. A comparative study between *Guadua angustifolia* Kunth and the *Phyllostachys pubescens* Mazel (referred to as Moso) bamboo indicates that the *Guadua* culm possesses a higher Young's modulus per density than the Moso [22].

The study also determined the MFA to be 8.3° for both species. This indicates that the *Phyllostachys*, a temperate bamboo, might have a generally lower l/d than the tropical species, leading to a lower strength in this research. The effect on stiffness is smaller, as to be expected from short fibre mechanics. This gives an indication that species grown in less optimal growing conditions (temperate climate) might develop into less optimal structures, shown by the shorter l/d ratio of the species. This hypothesis is worth further evaluation in future research.

3.4 Influence of chemical composition

Table 3 lists the results of the chemical characterization of the different species. It is assumed that the extractives and ash content do not contribute to the mechanical performance of the bamboo fibres. Therefore the results have been rescaled, so that the sum of the cellulose, hemicellulose and lignin content is 100%. A two sided t-test ($\alpha=0.05$) revealed that there is no significant difference in lignin content between the two considered ages for the GAK. For the DMM and PNB an increase in lignin content is seen of around 5%. Since for DMM and PNB younger ages are compared, it is concluded that the lignin content in the fibre is still changing in the first three years of growth.

For the cellulose content no significant difference between the GAK samples can be found, but between DMM 1 and DMM 3 and PNB 0.75 and PNB 2-4 a significant difference is found. The species that show an increase of lignin content when aging show a decrease of cellulose content. This is biased by the analysis method, that does not take into account voids that might get filled up with material while the plant ages. It is known that lignin develops through the years, known as lignification, and fills voids that are still present in the plant. However for the analysis, the fibres are milled, and the cell wall material is taken as 100%. Therefore, a decrease in cellulose does not necessarily mean that less cellulose is present, but in this case gives indication that the lignin to cellulose ratio is changing.

It is commonly accepted that cellulose is the main contributor to the stiffness and strength of the fibres. To investigate this effect, the IGBT results are considered, which give an average value of the mechanical properties of the fibres. Looking at the stiffness from the IGBT, 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 IGBT, 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. However, literature does not give any indication of a relationship between the two [12].

A plot of the mechanical properties (Young's modulus and strength) in function of the cellulose content was not made because of the previously mentioned bias on the cellulose results. The authors want to emphasise that a chemical composition determination based on weight fraction cannot help to explain mechanical properties that are related to volume dimensions. However, with this chemical analysis it is shown that lignification might take up to 3 years, but barely effects the stiffness of the *technical* fibres.

Comparing the cellulose content of the youngest age of each species with the determined elementary fibre l/d ratio, it is found that they are linearly related. A higher l/d ratio, also shows a higher cellulose content. At the present moment there seems to be no direct logical explanation for this, but the observation is made.

4 Conclusions

Three different bamboo species, each of two different ages were investigated. From the microscopic images it could be seen that the *Dendrocalamus membranaceus* Munro has a 10-25 % higher percentage of fibre bundles present then the *Guadua angustifolia* Kunth and the *Phyllostachys nigra* Boryana that was considered.

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

To investigate the mechanical properties, fibre tensile tests and impregnated fibre bundle test (IFBT) were performed. In contrast to other natural fibres, the results of the back calculated properties are in agreement with the results of the *technical* fibre tests. The hypothesis is that the high lignin content present in the bamboo fibre, prevents sliding of the *elementary* fibres in a *technical* fibre tensile test. Therefore a *technical* fibre tensile test is a good method to screen the properties of bamboo fibres. It should be noted that the absolute values of the modulus and strength in this research are lower than previous work [38], due to the extraction out of rewetted material.

From the IFBT it is concluded that the stiffness of bamboo fibres from the tropical species considered in this research is unaffected by the species nor the age of the plant. Even the modulus of the species grown in Europe is only less than 15% lower. The strength seems to be more affected by the place of growth, since the European bamboo considered, shows lower properties (30% lower). These mechanical properties are reflected in the *elementary* fibre dimensions, as the length over diameter ratio of tropical species has *elementary* fibre l/d ratios of at least 50 % higher, leading to higher mechanical properties.

Chemical analysis was performed to investigate the effect of age on the lignification and the relationship with the mechanical properties. From the research on the chemical composition of the fibres it seems that the lignin content is still changing during the first 3 years of growth. Though this does not have an effect on the mechanical properties considered. Therefore it is decided that the *technical* fibres of the bamboo plant can be considered mature, within one growing season.

Acknowledgements

The authors would like to acknowledge the Impulse Fund KU Leuven (BOF/IMP14/034) for the support.

References

- [1] Perremans D, Trujillo E, Ivens J, Van Vuure AW. Effect of discontinuities in bamboo fibre reinforced epoxy composites. *Composites Science and Technology*. 2018;155:50-7.
- [2] Müssig J. *Industrial Applications of Natural Fibres*. West Sussex: John Wiley & Sons Ltd; 2010.
- [3] Campilho RDSG. *Introduction to natural fiber Composites*. Natural fiber composites. Boca Raton, Florida, USA: CRC Press Taylor & Francis Group; 2016.
- [4] John MJ, Sabu T. *Natural Polymers Volume 1 : Composites*. Cambridge: The Royal Society of Chemistry; 2012.
- [5] Bourmaud A, Beaugrand J, Shah DU, Placet V, Baley C. Towards the design of high-performance plant fibre composites. *Progress in Materials Science*. 2018;97:347-408.
- [6] Lobovikov M, Paudel S, Piazza M, Ren H, Wu J. *World bamboo resources*. Rome, Italy: Food and Agriculture Organization (FAO); 2007.
- [7] Ohrnberger D. *The Bamboos of the World: Bambus Buch*; 1996.
- [8] Trujillo E. *Polymer composite materials based on bamboo fibers [PhD thesis]*. Leuven: KU Leuven; 2014.
- [9] Minke G. *Building with Bamboo: design and Technology of a Sustainable Architecture*. Basel, Switzerland: Birkhäuser Verlag GmbH; 2016.
- [10] Londoño XC, G.C.; Riaño, N.M.; López, Y. Characterization of the anatomy of *guadua angustifolia* (Poaceae: Bambusoideae) culms. *Bamboo Science and Culture: The Journal of the American Bamboo Society*. 2002;16(1):18-31.
- [11] van der Lugt P. *Booming Bamboo*. Naarden, The Netherlands: Materia Exhibitions B.V.; 2017.
- [12] Liese W, Köhl M. *Bamboo The plant and its uses*: Springer International Publishing Switzerland 2015.
- [13] Gielis J. *Future possibilities for bamboo in European agriculture*. Short report "Bamboo for Europe Project": Oprins Plant, 2000, Rijksoorsel, Belgium; 2000.
- [14] Yiping L, Yanxia L, Buckingham K, Henley G, Guomo Z. *Bamboo and Climate Change Mitigation*. International Network for Bamboo and Rattan (INBAR); 2010.
- [15] Zakikhani P, Zahari R, Sultan MTH, Majid DL. Extraction and preparation of bamboo fibre-reinforced composites. *Materials & Design*. 2014;63:820-8.
- [16] van Vuure AW, Osorio L, Trujillo E, Fuentes CA, Verpoest I. Long Bamboo Fibre composites. 18th International Conference on Composite Materials. Jeju Island, Korea 2011.
- [17] Trujillo E, Moesen M, Osorio L, Van Vuure AW, Ivens J, Verpoest I. Bamboo fibres for reinforcement in composite materials: Strength Weibull analysis. *Composites Part a-Applied Science and Manufacturing*. 2014;61:115-25.
- [18] Osorio L, Trujillo E, Van Vuure AW, Verpoest I. Morphological aspects and mechanical properties of single bamboo fibers and flexural characterization of bamboo/epoxy composites. *Journal of Reinforced Plastics and Composites*. 2011;30(5):396-408.
- [19] Lefeuvre A, Bourmaud A, Morvan C, Baley C. Elementary flax fibre tensile properties: Correlation between stress-strain behaviour and fibre composition. *Industrial Crops and Products*. 2014;52:762-9.

- [20] Marrot L, Lefeuvre A, Pontoire B, Bourmaud A, Baley C. Analysis of the hemp fiber mechanical properties and their scattering (Fedora 17). *Industrial Crops and Products*. 2013;51:317-27.
- [21] Lybeer B, Koch G, Van Acker J, Goetghebeur P. Lignification and cell wall thickening in nodes of *Phyllostachys viridiglaucens* and *Phyllostachys nigra*. *Annals of Botany*. 2006;97(4):529-39.
- [22] Dixon PG, Ahvenainen P, Aijazi AN, Chen SH, Lin S, Augusciak PK, Borrega M, Svedstrom K, Gibson LJ. Comparison of the structure and flexural properties of Moso, Guadua and Tre Gai bamboo. *Construction and Building Materials*. 2015;90:11-7.
- [23] Wang X, Ren H, Zhang B, Fei B, Burgert I. Cell wall structure and formation of maturing fibres of moso bamboo (*Phyllostachys pubescens*) increase buckling resistance. *Journal of the Royal Society Interface*. 2012;9(70):988-96.
- [24] Grosser D, Liese W. On the anatomy of Asian bamboos, with special reference to their vascular bundles. *Wood Science and Technology*. 1971;5(290).
- [25] Liese W. *The Anatomy of Bamboo Culms: International Network for Bamboo and Rattan*; 1998.
- [26] Crow E, Murphy RJ. Microfibril orientation in differentiating and maturing fibre and parenchyma cell walls in culms of bamboo (*Phyllostachys viridi-glaucens* (Carr.) Riv. & Riv.). *Botanical Journal of the Linnean Society*. 2000;134(1):339-59.
- [27] Ren D, Yu Z, Li W, Wang H, Yu Y. The effect of ages on the tensile mechanical properties of elementary fibers extracted from two sympodial bamboo species. *Industrial Crops and Products*. 2014;62:94-9.
- [28] Wang HK, Tian GL, Li WJ, Ren D, Zhang XX, Yu Y. Sensitivity of bamboo fiber longitudinal tensile properties to moisture content variation under the fiber saturation point. *Journal of Wood Science*. 2015;61(3):262-9.
- [29] Parameswaran N, Liese W. On the fine structure of bamboo fibres. *Wood Science and Technology*. 1976;10(231).
- [30] Bourmaud A, Morvan C, Bouali A, Placet V, Perre P, Baley C. Relationships between micro-fibrillar angle, mechanical properties and biochemical composition of flax fibers. *Industrial Crops and Products*. 2013;44:343-51.
- [31] Yu Y, Wang HK, Lu F, Tian GL, Lin JG. Bamboo fibers for composite applications: a mechanical and morphological investigation. *Journal of Materials Science*. 2014;49(6):2559-66.
- [32] Gritsch CS, Murphy RJ. Ultrastructure of fibre and parenchyma cell walls during early stages of culm development in *Dendrocalamus asper*. *Annals of Botany*. 2005;95(4):619-29.
- [33] Itoh T. Lignification of Bamboo (*Phyllostachys heterocycla* Mitf.) During its Growth. *Holzforschung*. 1990;44(3):191-200.
- [34] Murphy RJ, Alvin KL. Fibre maturation in the bamboo *Gigantochloa scortechinii*. *Iawa Journal*. 1997;18(2):147-56.
- [35] Gritsch CS, Kleist G, Murphy RJ. Developmental changes in cell wall structure of phloem fibres of the Bamboo *Dendrocalamus asper*. *Annals of Botany*. 2004;94(4):497-505.
- [36] Li XBS, T.F.; Peter, G.F.; Hse, C.Y.; Eberhardt, T.L. Chemical changes with maturation of the bamboo species *phyllostachys pubescens*. *Journal of Tropical Forest Science*. 2007;19(1):6-12.
- [37] Lin JX, He XQ, Hu YX, Kuang TY, Ceulemans R. Lignification and lignin heterogeneity for various age classes of bamboo (*Phyllostachys pubescens*) stems. *Physiologia Plantarum*. 2002;114(2):296-302.
- [38] Depuydt D, Hendrickx K, Biesmans W, Ivens J, Van Vuure AW. Digital image correlation as a strain measurement technique for fibre tensile tests. *Composites Part A: Applied Science and Manufacturing*. 2017;99:76-83.
- [39] Bui DT, Pradhan B, Lofman O, Revhaug I, Dick OB. Regional prediction of landslide hazard using probability analysis of intense rainfall in the Hoa Binh province, Vietnam. *Natural Hazards*. 2013;66(2):707-30.
- [40] KMI. Huidig Klimaat België. <https://www.meteo.be/meteo/view/nl/6042923-Huidig+klimaat+Belgie.html>.
- [41] Hendrickx K, Romian R, Goedemé T, Van Vuure A, Ivens J. Rapid and effective methods for the screening of flax fibres for composite applications. *Proc International Conference on Composite Materials (ICCM)*. Copenhagen, Denmark. 2015;20 (1).

- [42] Bensadoun F, Verpoest I, Baets J, Müssig J, Graupner N, Davies P, Gomina M, Kervoele A, Baley C. Impregnated fibre bundle test for natural fibres used in composites. *Journal of Reinforced Plastics and Composites*. 2017;0731684417695461.
- [43] Shah DU, Nag RK, Clifford MJ. Why do we observe significant differences between measured and 'back-calculated' properties of natural fibres? *Cellulose*. 2016;23(3):1481-90.
- [44] Franklin GL. Preparation of Thin Sections of Synthetic Resins and Wood-Resin Composites, and a New Macerating Method for Wood. *Nature*. 1945;155:51.
- [45] Vazquez-Cooz I, Meyer RW. A differential staining method to identify lignified and unlignified tissues. *Biotechnic & Histochemistry*. 2002;77(5-6):277-82.
- [46] Sweygers N, Somers MH, Appels L. Optimization of hydrothermal conversion of bamboo (*Phyllostachys aureosulcata*) to levulinic acid via response surface methodology. *Journal of Environmental Management*. 2018;219:95-102.
- [47] Walker JCF. *Primary Wood Processing*. Dordrecht, The Netherlands: Springer 2006.
- [48] Defoirdt N, Biswas S, De Vriese L, Tran LQN, Van Acker J, Ahsan Q, Gorbatikh L, Van Vuure A, Verpoest I. Assessment of the tensile properties of coir, bamboo and jute fibre. *Composites Part a-Applied Science and Manufacturing*. 2010;41(5):588-95.
- [49] Rao KMM, Rao KM. Extraction and tensile properties of natural fibers: Vakka, date and bamboo. *Composite Structures*. 2007;77(3):288-95.
- [50] Okubo K, Fujii T, Yamamoto Y. Development of bamboo-based polymer composites and their mechanical properties. *Composites Part a-Applied Science and Manufacturing*. 2004;35(3):377-83.
- [51] Berges M, Leger R, Placet V, Person V, Corn S, Gabrion X, Rousseau J, Ramasso E, Ienny P, Fontaine S. Influence of moisture uptake on the static, cyclic and dynamic behaviour of unidirectional flax fibre-reinforced epoxy laminates. *Composites Part a-Applied Science and Manufacturing*. 2016;88:165-77.
- [52] Shah DU. Damage in biocomposites: Stiffness evolution of aligned plant fibre composites during monotonic and cyclic fatigue loading. *Composites Part a-Applied Science and Manufacturing*. 2016;83:160-8.
- [53] Blanchard JMFAS, A.J.; Blake, J.I.R. Assessing the feasibility of natural composite for structural applications. *ECCM17*. Munich. 2016.
- [54] Oksman K, Wallstrom L, Berglund LA, Toledo RD. Morphology and mechanical properties of unidirectional sisal-epoxy composites. *Journal of Applied Polymer Science*. 2002;84(13):2358-65.
- [55] Charlet K, Baley C, Morvan C, Jernot JP, Gomina M, Breard J. Characteristics of Hermes flax fibres as a function of their location in the stem and properties of the derived unidirectional composites. *Composites Part a-Applied Science and Manufacturing*. 2007;38(8):1912-21.
- [56] Osorio L, Trujillo E, Lens F, Ivens J, Verpoest I, Van Vuure AW. In-depth study of the microstructure of bamboo fibres and their relation to the mechanical properties. *Journal of Reinforced Plastics and Composites*. 2018;0731684418783055.

Figure 1 Exploded view of the hierarchical structure of a bamboo plant, from culm to microfibril, incorporating the polylamellate elementary fibre structure according to Parameswaran and Liese [29]

Figure 2 Four basic types of vascular bundles. Type I consists out of one part, the central vascular bundle with a supporting tissue of four sclerenchyma sheaths; Type II is similar to Type I but with the inner bundle sheath at the protoxylem strikingly larger; Type III consists out of two parts, besides the central vascular bundle with the four sclerenchyma sheaths an additionally one isolated fibre strand is present; Type IV consists out of three parts, the central vascular strand with smaller sclerenchyma sheaths and two isolated fibre strands. Reprinted from “On the Anatomy of Asian Bamboos with Special Reference to their Vascular Bundles, D. Grosser & W. Liese, 1971, with permission from Springer Nature” [24]

Figure 3 Overview of the natural distribution of bamboo and the different genera used in this research, with the red dots indicating the place of harvest of the considered species. Reprinted from “The Bamboos of the World, D. Ohrnberger, 1998, with permission from Elsevier” [7]

Figure 4 Cross section of the considered bamboo species: *Guadua angustifolia* Kunth (GAK); *Dendrocalamus membranaceus* Munro (DMM); *Phyllostachys nigra* Boryana (PNB), with the numbers referring to the age in years. Scale 1000 μm .

Figure 5 Analysis on the cross section of the bamboo culm DMM1 of the areal fibre bundle fraction, with the contour of the fibre sheaths and strands indicated. The area enclosed by the contours was used to calculate the areal fraction. Scale 1000 μm .

Figure 6 Diameter distribution of the different species and ages, *Guadua angustifolia* Kunth (GAK), *Dendrocalamus membranaceus* Munro (DMM), *Phyllostachys nigra* Boryana (PNB)

Figure 7 Scanned images used as input for the fibre diameter characterisation of the extracted fibres with a) GAK 1-3, b) DMM 1 and c) PNB 0.75.

Figure 8 Comparison results of the a) Young's modulus and the b) ultimate tensile strength from the fibre tensile test and the back calculated properties from the impregnated fibre bundle test (IFBT), c) the strain to failure of the fibre tensile test and the composite failure strain measured in the impregnated fibre bundle test (IFBT).

Figure 9 Plot of the tensile strength and Young's modulus versus the *technical* fibre diameter of the fibre tensile test results.

Figure 10 One typical stress strain curve of the tested composites via IFBT for each species and age considered in this research.

Figure 11 *Elementary* fibres after maceration were prepared on glass slides and scanned a) GAK 1-3, b) DMM 1, c) PNB 0.75, d) diameter characterization by light microscope.

Figure 12 *Elementary* fibre diameter versus fibre length plot for the three considered species, the youngest age.

Table 1 Overview of the test samples for the fibre tensile test and the IGBT.

Material	Fibre density (g/cm ³)	Average diameter (μ m)	# specimens Fibre tensile test	# specimens IGBT
GAK 1-3	1.41 $\pm$ 0.03	185 $\pm$ 63	18	5
GAK 3-5	1.44 $\pm$ 0.01	144 $\pm$ 31	17	6
DMM 1	1.41 $\pm$ 0.02	134 $\pm$ 23	13	5
DMM 3	1.42 $\pm$ 0.02	228 $\pm$ 75	13	4
PNB 0.75	1.42 $\pm$ 0.02	177 $\pm$ 50	16	4
PNB 2-4	1.38 $\pm$ 0.01	167 $\pm$ 36	18	5

Table 2 Summary of the results of the IGBT.

Material	Back calculated Young's modulus (GPa)	Back calculated fibre strength (MPa)	Composite failure strain (%)
GAK 1-3	44 $\pm$ 2	481 $\pm$ 93	1.06 $\pm$ 0.06
GAK 3-5	41 $\pm$ 3	426 $\pm$ 41	1.22 $\pm$ 0.34
DMM 1	41 $\pm$ 3	436 $\pm$ 62	0.98 $\pm$ 0.08
DMM 3	41 $\pm$ 1	380 $\pm$ 28	0.93 $\pm$ 0.06
PNB 0.75	37 $\pm$ 3	293 $\pm$ 19	0.77 $\pm$ 0.04
PNB 2-4	35 $\pm$ 3	329 $\pm$ 31	0.90 $\pm$ 0.10

Table 3 Results of the chemical characterization of the considered species.

Material	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives (g/100g)	Ash (g/100g)
GAK 1-3	54.6 ± 1.2	14.0 ± 0.3	31.4 ± 1.0	0.7 ± 0.5	0.6 ± 0.1
GAK 3-5	53.7 ± 0.2	15.2 ± 0.5	31.0 ± 0.1	0.5 ± 0.4	5.0 ± 0.4
DMM 1	58.5 ± 0.4	15.3 ± 0.3	26.7 ± 0.4	0.0 ± 0.3	0.3 ± 0.2
DMM 3	50.4 ± 0.3	16.8 ± 0.1	32.8 ± 0.7	9.7 ± 0.6	0.1 ± 0.0
PNB 0.75	52.8 ± 1.0	17.9 ± 1.0	29.3 ± 0.5	7.9 ± 0.6	0.2 ± 0.0
PNB 2-4	49.2 ± 0.2	16.5 ± 0.1	34.2 ± 0.4	4.7 ± 0.4	0.5 ± 0.1