

# The potential of European bamboo fibre for composite applications: a chemical-mechanical approach.

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## ABSTRACT

The presented work investigates the tensile properties as well as the sorption behaviour and chemical composition of European bamboo fibres. Single fibre test results show that there might be an influence of the time of harvesting on the tensile properties after fibre extraction. The sorption behaviour is in line with other natural fibres, taking the chemical composition into account.

## INTRODUCTION

European bamboo fibres have not yet been investigated in literature, although several winter hard species exist, making them potentially interesting for the European composite industry. For this research, the *Phyllostachys Nigra Boryana* was selected, due to its high yield and its hardness necessary to withstand the European climate. This study aims to reveal the tensile and sorption behaviour of the fibres and link it with the chemical fibre composition.

## MATERIALS AND METHODS

Bamboo fibres were extracted from the bamboo species *Phyllostachys Nigra Boryana* grown in Belgium on a latitude of 50,95° and longitude of 5,41°. Belgium has a temperate climate with an average annual temperature of 9.8°C and an average relative humidity of 80% with an annual rainfall of 800 mm [1]. Two to three culms were harvested for each selected age, i.e. 9 months and older than 24 months, after which fibres were extracted from the middle region of the plant. The 9 month year old plant will be referred to as B9 and the plants older than 2 years as B24.

Fibre tensile tests were performed according to ASTM C1557 with an Instron 5943. Fibre samples were prepared with a gauge length of 5 cm and optical flags attached to the fibre, in order to measure the strain via digital image correlation. A crosshead displacement of 1.5mm/min was maintained and data was collected every 500 ms. The fibres were pneumatically clamped at 6 bar using rubber-faced clamps of 10 x 30 mm. The force was registered with a 1kN load cell and the cross sectional area of the fibre was calculated from the mass and the fibre density. All tests were performed in a conditioned room with a relative humidity of 50% and an average temperature of 21°C. Fibres were at least for 3 days prior to testing conditioned in that room.

Gas pycnometer measurements were performed to determine the density of the fibres, using a Beckman air comparison pycnometer model 930. The fibres were milled prior to measuring and had a particle size smaller than 250  $\mu\text{m}$ .

Dynamic vapour sorption (DVS) measurements were performed to investigate the moisture uptake capacity of the fibres at different levels of relative humidity (RH). The equipment consists of a Cahn microbalance, a temperature-controlled housing and mass flow controllers that control the appropriate flow of the wet and dry  $\text{N}_2$  gas. The benefit of this technique is the control over both the relative humidity and the temperature (i.e. 21°C). The fibres were milled prior to the test as described for the gas pycnometer measurement. Approximately 5-10 mg of the bamboo fibre dust powder was used. The sample was first dried to a relative humidity (RH) of 0%; subsequently, the humidity was changed to different levels (30, 60 and 90%). Every subsequent step was initiated when the change of the sample mass as a function of time was lower than 0.02 mg/min. A desorption curve with equal levels of RH was realized till full desorption. The moisture content was calculated according to the following equation:

$$\text{Water content} = \frac{m_{eq} - m_d}{m_d}$$

Where  $m_{eq}$  is the mass of the sample at equilibrium and  $m_d$  the mass of the dry sample reached after the first drying stage.

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. (2016) was used. In a first step, a neutral detergent solvent (Na-lauryl sulphate, EDTA, pH = 7) was applied, thereby extracting the digestible cell contents (soluble sugars, starch) while the hemicellulose, cellulose and lignin fractions remain. In a second step, an acidic detergent solvent was applied (1 M  $\text{H}_2\text{SO}_4$ ) to extract hemicellulose, while cellulose and lignin remain as a solid residue. In a third step concentrated sulfuric acid (72 w%) was applied to extract cellulose, whereby lignin remains as the last cell wall component. The determination of the Klason lignin content was performed according to the methodology described in the handbook of wood chemistry and wood composites [2].

## RESULTS AND DISCUSSION

### Single fibre test

From the gas pycnometer measurement the density of the bamboo fibres from the *Phyllostachys Nigra* Boryana could be determined to be  $1.41 \pm 0.02 \text{ g/cm}^3$ . No distinction in age could be found. This value is in agreement with previous research on the *Guadua Angustifolia* Kunth, which showed a fibre density of  $1.4 \text{ g/cm}^3$  [3]. The fibre density is used to calculate the cross sectional area of the bamboo fibres necessary for the tensile test.

The results of the tensile test are displayed in Figure 1. Fibres harvested in February of the year 2016, showed no difference in mechanical properties for a different age. A student's t-test was performed to investigate the difference in the means of the Young's modulus, the strength and the strain to failure. For the Young's modulus and strength no significant difference could be found between 9B and 24B fibres. The modulus of the 9B fibres is  $35 \pm 12 \text{ GPa}$  and for the 24B it is  $33 \pm 11 \text{ GPa}$ . The respective strength is  $239 \pm 155 \text{ MPa}$  for 9B and  $297 \pm 130 \text{ MPa}$  for 24B. The strain to failure of 24B is 30% larger than the strain to failure of the 9B bamboo fibres, respectively  $1.1 \pm 0.4 \%$  versus  $0.8 \pm 0.3 \%$ . The average fibre diameter of 9B fibres is  $215 \pm 48 \mu\text{m}$ , for 24B  $240 \pm 48 \mu\text{m}$ .

However, a difference could be found between the properties of the fibres of the same bamboo species, harvested at a different time. Fibres harvested in June 2015 had a Young's modulus of  $53 \pm 10$  GPa, which is in the same order as the results on the *Guadua Angustifolia* Kunth [3], and a tensile strength of  $483 \pm 177$  MPa with a strain to failure of  $1.0 \pm 0.4$  %. From literature it is known that the bamboo plants in a European climate contain half as much of water during autumn and spring as compared to summer [4]. From this a hypothesis can be derived that fibre extraction in the summer months introduces less damage to the fibres and thus leads to better mechanical properties. This will be further investigated.

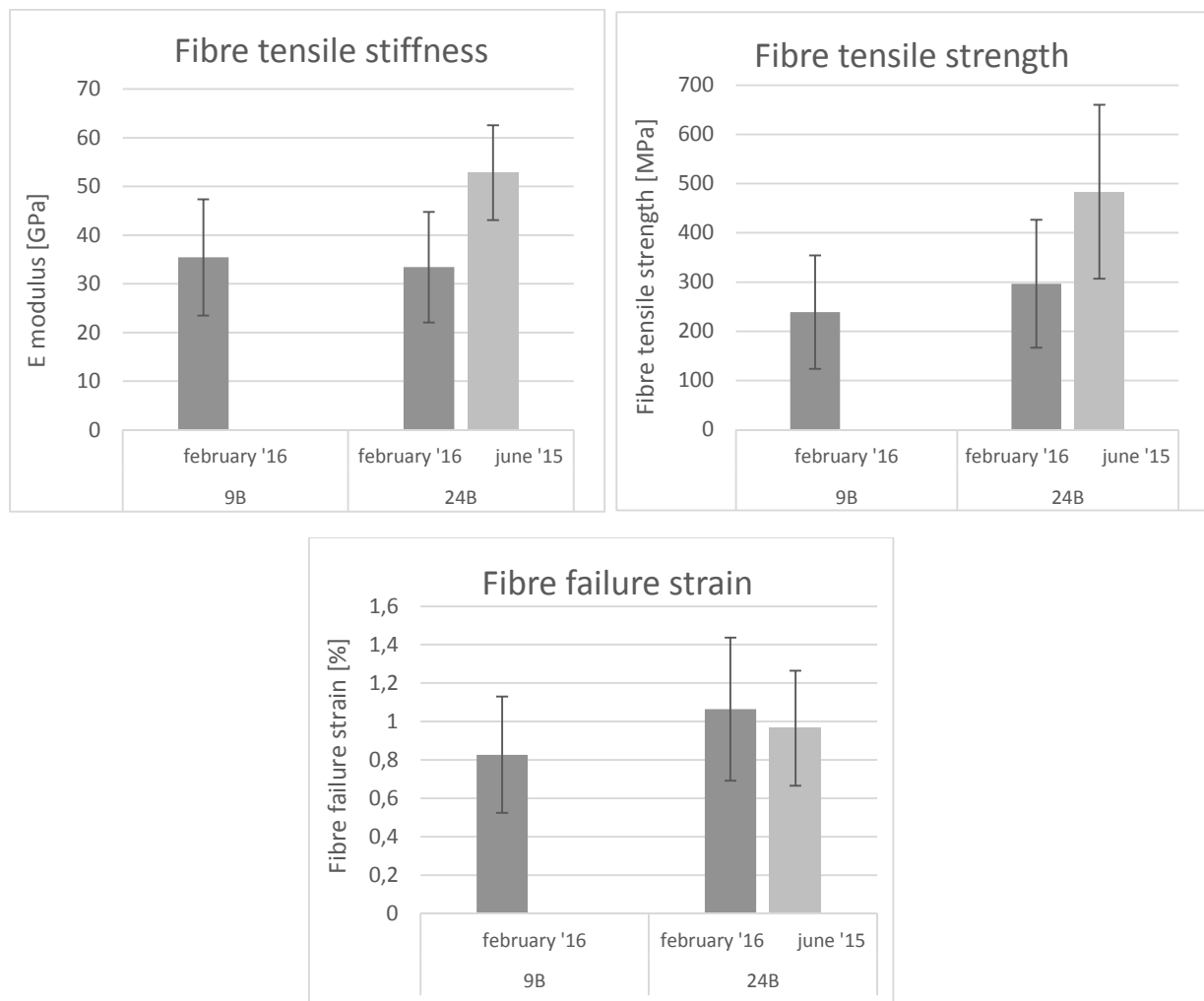


Figure 1 Results of the fibre tensile tests with a) the tensile modulus, b) the tensile strength, c) the strain to failure.

### Dynamical vapour sorption

The sorption isotherms measured by DVS are shown in Figure 2. Up to 60% RH both the isotherm for the 9B and 24B show similar behaviour. For high relative humidity a distinction can be found, although caution should be taken analysing the data above 70% RH. Hill et al. [5] have shown that the reproducibility of sorption tests for natural fibres above a relative humidity of 70% deteriorates [5]. Therefore further investigation should indicate whether young fibres really absorb more moisture at high relative humidity.

When comparing the sorption data for bamboo with the data on other natural fibres obtained by Hill et al. (paper of 2009 figure 5), a good similarity in data can be found. The curve of the equilibrium moisture content of bamboo fibre lies above the one for flax fibre and right below the curve for coir fibres. It is known that the accessibility of OH groups determines for a large amount the moisture uptake of the fibre. However, looking at the amount of hemicellulose alone, does not explain the results in literature [5]. Therefore Hill et al. assume that also the lignin content plays a role. In this way the high moisture uptake for coir fibres is explained by the ability of lignin to accommodate water within the cell walls. Another hypothesis that is worthwhile investigating is the contribution of the amount of amorphous cellulose, which is known to be the second most sensitive component to water uptake [6].

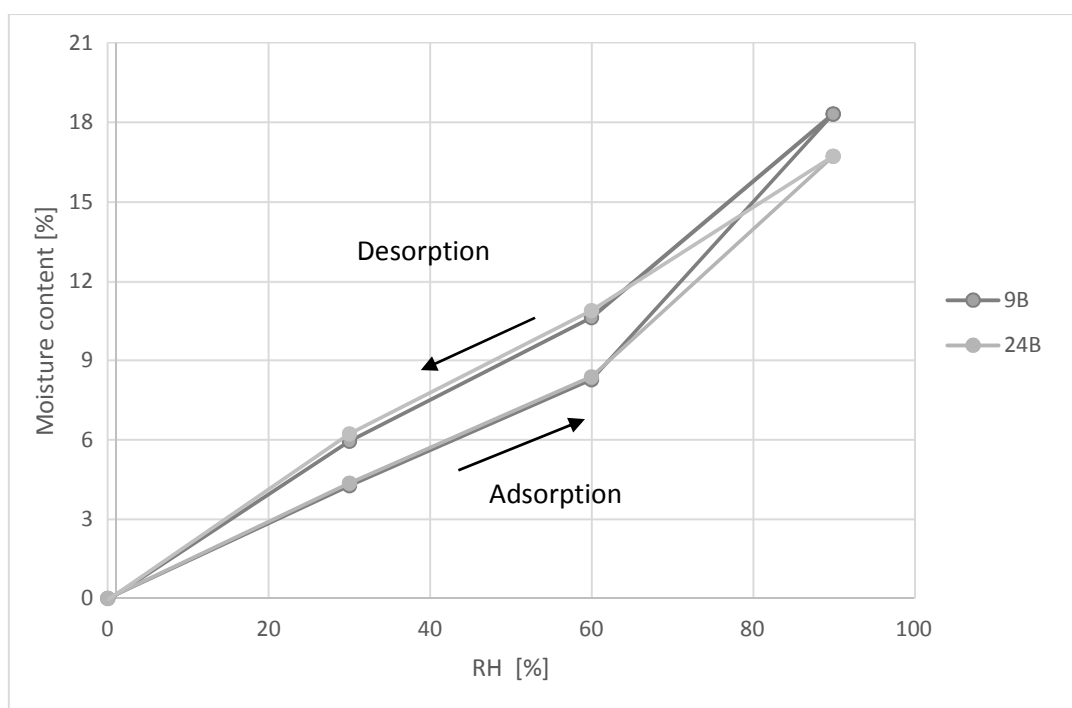


Figure 2 Moisture content as a function of the relative humidity for the bamboo fibres 9B and 24B harvested in February '16.

### Chemical composition

The chemical composition determined by the adapted van Soest method is shown in Table 1. Performing a student's t-test, no significant difference can be found between the hemicellulose, cellulose and lignin content of the B9 and B24 bamboo fibres. The results of the lignin content are rather high compared to the bulk bamboo material in literature; therefore, a Klason lignin test was performed as a form of control.

The results of the Klason lignin test showed no significant difference between B9 and B24. However the lignin content was 23% lower compared to the adapted Van Soest method. In the first test, the small particle size of the bamboo fibres that were milled caused an agglomeration of bamboo dust that inhibited the chemicals to reach all the particles. Therefore, it is highly likely that the first method gives an underestimation of the cellulose and maybe hemicellulose content, causing an overestimation of the lignin content. The Klason lignin values are thus considered as representative lignin quantities for the material.

Compared to other natural fibres, bamboo has a lignin content lower than coir (45%), but higher than jute (13%) and flax (3%). Linking the chemical composition with the sorption data, further investigation is needed to clarify whether the lignin content or the amorphous cellulose content determine the sorption behaviour the most.

Comparing the bamboo bulk material, bamboo fibres possess more lignin. Li et al. [7] registered an average Klason lignin content for the *Phyllostachys Pubescens* culm of 1 to 3 years of 23%. This is in the same order as Itoh et al. [8] who found an average lignin content for the *Phyllostachys heterocycla* of 2 to 3 years old bamboo of 26%. This would mean that the fibres measured in this study have up to 40% more lignin compared to the bulk material mentioned in literature. Further investigation will be performed on the bulk composition of the European *Phyllostachys Nigra* Boryana to make final conclusions.

Table 1 Chemical fibre composition of the bamboo B9 and B24 fibres. The fibres were milled prior to the determination of the composition.

| Material | Adapted van Soest  |                              |                          |                       |                    | Klason lignin         |
|----------|--------------------|------------------------------|--------------------------|-----------------------|--------------------|-----------------------|
|          | NDF [%]<br>(stdev) | Hemicellulose<br>[%] (stdev) | Cellulose<br>[%] (stdev) | Lignin [%]<br>(stdev) | Ash [%]<br>(stdev) | Lignin [%]<br>(stdev) |
| B9       | 12.42 (0.30)       | 22.18 (0.63)                 | 20.33 (0.39)             | 44.29 (1.03)          | 0.45 (0.07)        | 34.02 (1.11)          |
| B24      | 9.68 (0.39)        | 21.70 (0.65)                 | 20.21 (1.19)             | 46.36 (1.02)          | 0.04 (0.04)        | 35.99 (0.55)          |

## CONCLUSION

This paper has investigated the potential of European bamboo. Focus was put on the mechanical properties, sorption behaviour and chemical composition.

Tensile fibre tests have shown that there might be an influence of the harvesting season. Fibres harvested in June had similar properties as reported for the South American *Guadua Angustifolia* Kunth fibres [3].

The sorption isotherm shows good agreement with the trend for other natural fibres measured by Hill et al. [5].

Care should be taken when determining the fibre composition via the Van Soest method, since particle size might influence the accuracy of the data. The lignin content was obtained via the Klason lignin method and indicates that the fibres are richer in lignin than the bulk bamboo material described in literature [7,8].

## REFERENCES

- [1] KMI. "General climate Belgium" Internet: [www.meteo.be/meteo/view/nl/6042922-Algemeen+klimaat+Belgie.html](http://www.meteo.be/meteo/view/nl/6042922-Algemeen+klimaat+Belgie.html), [Date accessed Apr. 22, 2016]
- [2] R.M. Rowell. *Handbook of wood chemistry and wood composites*. Florida: CRC Press, 2005
- [3] L. Osorio et al. "Morphological aspects and mechanical properties of single bamboo fibers and flexural characterization of bamboo/epoxy composites". *Reinforced plastics & composites*, vol. 30 (5), pp. 396-408, 2011
- [4] D. Van Goethem. "Growth and ecophysiological characterisation of a *Phyllostachys humilis* bamboo plantation in Ireland" PhD dissertation, University of Antwerp, Antwerp, 2013

[5] C.A.S. Hill et al. "The water vapor sorption behaviour of natural fibers". *Journal of Applied polymer science*, vol. 112 (3), pp. 1524-1537, 2009

[6] C. Baillie. *Green composites, polymer composites and environment*. Cambridge: Woodhead Publishing Limited, 2004

[7] X.B. Li et al. "Chemical changes with maturation of the bamboo species *phyllostachys pubescens*". *Journal of Tropical Forest Science*, vol.19 (1), pp. 6-12, 2007

[8] T. Itoh. "Lignification of bamboo (*Phyllostachys heterocycla* Mitf.) during its growth". *Holzforschung*, vol. 44 (3), pp. 191-200, 1990