



Evaluation of Bast Fibres of the Stem of *Carica papaya* L. for Application as Reinforcing Material in Green Composites

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Authors' contributions

This work was carried out in collaboration between all authors. Authors AK and CN wrote the paper and concept of experiment. Author AG execution of mechanical tests and optical measurements. Author TL determination of density, supervision. Author AR determination of lignin, cellulose and hemicelluloses contents. Author ME determination of microfibril angle. All authors read and approved the final manuscript.

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ABSTRACT

Aims: The production of green composites based on natural fibres is rising with regard to increasing environmental problems and declining fossil raw materials. Bast fibres of papaya (*Carica papaya* L.) accumulate on plantations at a large scale but remain an unused resource. The characterisation of the bast allows a first evaluation of the potential of papaya-fibres for use in composites.

Study Design: Material testing.

Place and Duration of Study: Institute for Botany, Technische Universität Dresden and

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Methodology: The anatomic structure of fibre cells and the microfibril angle of the cell walls were determined as well as a chemical analysis to determine the proportion of cellulose, lignin and hemicelluloses of the fibre cells. In addition, samples of fibres were subjected to static uniaxial tension tests revealing Young's modulus, tensile strength and breaking strain at different plant ages and of two origins.

Results: Fibres of a two-year-old plant exhibited a Young's modulus of 10.7 GPa, a tensile strength of 101 MPa and a breaking strain of 1.2%, on average. Fibres from six-months-old plants, grown under greenhouse conditions had a mean Young's modulus of 4.4 GPa, tensile strength of 49 MPa and a breaking strain of 1.4%. Having one of the lowest fibre densities with ca. 0.85 g/cm³, papaya fibres exhibit noteworthy specific mechanical properties among all studied natural fibers.

Conclusion: These data allow us a first estimation for a potential use in green composites as reinforcing material.

Keywords: Carica papaya; bast fibres; Young's modulus; fibre characterization.

1. INTRODUCTION

Composite materials are increasingly important for the production of light and stiff constructions for various applications. However, apart from the favourable mechanical properties, composites have some shortcomings. Primarily, the recycling of components turns out to be difficult and a large amount of composites end up in dumps or incinerators. Even though the energy value of the material is used, there is still a contribution to CO₂ emissions and environmental pollution [1]. Apart from that, the non-renewable resource mineral oil has to be used for some matrices and fibres, such as polymer matrices or carbon fibres. Therefore, alternatives are being highly looked after in view of dwindling resources and the increasing ecological awareness.

Plant fibres offer some advantages over synthetic materials. They provide carbon neutrality when being burned, which is highly relevant with regard to ecological problems. At the same time, they are less abrasive to production machinery due to their flexibility and perform as good acoustic and thermal isolators. Plant fibres are usually of low density and the costs of the fibres (on a volumetric basis) are low [1,2]. Still, there are disadvantages, especially the high variability of mechanical properties depending on the conditions of growth (nutrition, exposure to wind and sunlight) and the age of the plant [1,2]. Furthermore, the hydrophilic character of natural fibres may severely affect the material properties. Due to swelling of fibres upon moisture uptake and a reduction of the binding of fibre and matrix can be reduced, stiffness may decrease or the overall geometry may be altered. Additionally a maximum processing temperature must not be exceeded to

avoid damage to the fibre [1]. However, there is a broad field of application for bio-composites, for example in the production of casing structures or in automobile and packaging industries, as not for all components the mechanical properties of advanced composites are needed [1]. Precondition for the use of natural fibres in composites is a deep knowledge of their mechanical properties. The characterisation of fibres is the first step to evaluate their potential for use in composites. Knowledge of Young's modulus, strength and breaking strain allows the selection of suitable matrix material in order to achieve appropriate material properties of the composite. Mechanical properties of various natural fibres as flax, hemp, jute, sisal and more are already investigated and comprehensively summarised by Faruk et al. [2] and Muessig [3].

So far fibre properties of Papaya, another interesting resource of natural fibres, have not been studied yet. Papaya plants may reach an age of 20 years and do usually not branch [4]. Characteristically, the secondary xylem remains completely parenchymatous [5,6,7]. Despite the lack of wood, individual plants grow up to nine metres [5,7,8]; hence papaya is called a giant herb [4]. Lignified fibres occurring in the bark (secondary phloem) are the only reinforcing structures interacting with the turgor pressure [9]. The fibres form a complex lattice like mesh, which again is filled with parenchyma (Fig. 1). The small amount of fibres compared to the total weight of the stem arouses interest as a model for light-weight applications and structures. Papaya plants were cultivated primarily due to the palatable fruits. Main producers in 2012 have been India (5.16 million ton), Brazil (1.52 million ton) and Indonesia (0.91 million ton). The world's total yield in 2012 accounted for ca. 12.4 million

ton [10]. Papaya may therefore be an interesting source for fibres as by-product after the plants have been harvested, such as pineapple, oil palm and coir. Since the papaya fruit yield is at a maximum in the first three years, plants are replaced mostly after 3 - 5 years. At that point fibre material is available in a large scale that is usually discarded. Further processing of fibre material in composites could be an additional source of income for the producer. So far, the only idea for an application was the suggestion to use papaya fibres as biosorbent to remove heavy metals from water [11,12].

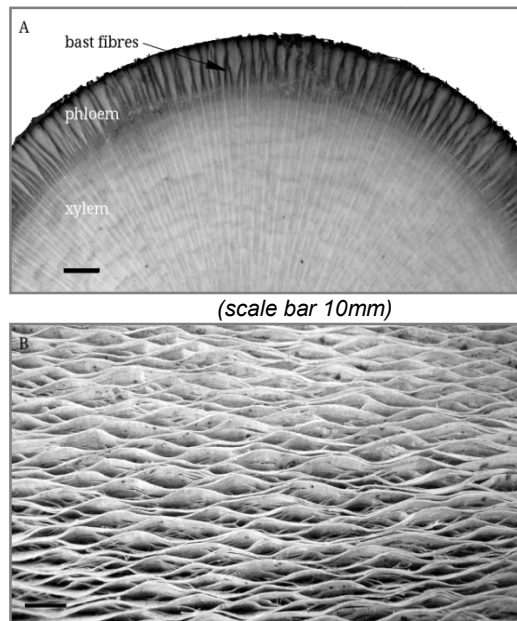


Fig. 1. Detail of papaya stem's cross section (A) and fibres after removal of parenchyma in longitudinal view (B)

2. MATERIALS AND METHODS

2.1 Materials

In a botanical definition a single cell is termed as fibre when the length is much larger than the diameter. In fibre technology usually bundles of individual fibre cells are processed. For simplification, the term fibre is used here for bundles of cells that in fact are lamellae.

We put emphasis on experimentally determining papaya fibre's Young's modulus, tensile strength and breaking strain by static tension tests. Fibres for mechanical analyses were taken from the base of plant between 0 and 10 cm above

ground. The fibre mesh was isolated by maceration in water. After one month of immersion non-lignified cells could easily be removed from the fibre mesh. Subsequently the fibre mesh was rinsed and cleaned with water and kept in water until testing. The mesh was cut in individual samples of ca. 3 cm in length and 0.1 - 1.1 mm width using a scalpel and dried at room temperature before testing. Fibre properties have been compared from plants of two different sources: 1. a commercial plantation in Nicaragua and 2. plants, grown in a greenhouse at the botany institute of the TU Dresden. In Nicaragua, samples were taken from a two year old papaya plantation (*Carica papaya* L. 'Red Lady') in November 2011. The plant had a height of two metres and a diameter of ca. 10 cm. In Dresden *Carica papaya* L. 'Red Lady' plants were grown from seeds in the greenhouses of the institute in April 2011. Samples for mechanical testing were taken from two of these plants, aged six months and with heights of ca. one metre and with a diameter of about one centimetre. Samples of another three plants were used for anatomical investigations.

2.2 Anatomy

Fibre dimensions were analysed using Scanning Electron Microscopy (SEM). Fibre cells were measured based on cross sections of three plants. Cell dimensions were calculated from the pixel number corresponding to cell length and width and a calibration image taken of a standardised scale with 1 μ m scaling, using GIMP, an open-source image editing program. Wide-angle X-ray diffraction (WAXD) has been used to measure average microfibril angles (MFA) of the cell wall according to Lichtenegger et al. [13].

2.3 Density, Contents of Cellulose, Hemicellulose and Lignin

The density of fibres was determined according to DIN EN ISO 1183-1 using a precision balance and a pycnometer with an accuracy of 0.01 g and 0.05 ml respectively. Cellulose, hemicellulose, and lignin content were determined according to Kuerschner [14], Poljak [15] and Klason [16]. Details of the extraction process are described by Bremer [17].

2.4 Mechanical Tests

All specimens were subjected to tension tests using a dynamic mechanical analyser (DMA TA

instruments Q800) at the Fraunhofer Institute for Non-destructive Testing Dresden (IZFP-D). Experiments were recorded with an force sensitivity of 0.01 mN and a displacement accuracy of 1 nm. The samples were strained at a rate of 1 N/min to reduce speed dependent influences. Since the fibres have a sheet like cross sectional area, the width was measured using a microscope at 25 - 50 fold magnification. The thickness was measured using a vernier calliper. Stress-strain diagrams were compiled using the program TA Universal Analysis 2000. Tension stiffness and Young's modulus were calculated from the slope of the initial linear part of the stress-strain curve. Additionally ultimate tensile strength (maximum stress that a material can withstand before breaking) and breaking strains (maximum strain when failure occurs) were determined.

2.5 Data Analysis

The obtained data were analysed by Mann Whitney U-test comparing differences of morphological and mechanical results with regard to origin of the samples. P values less than 0.05 were considered significant. Spearman rank correlation was used to check any correlation of morphological and mechanical results, i.e. whether morphological differences cause mechanical differences.

3. RESULTS

3.1 Anatomy

Fibre bundles contained between 15 and 130 individual fibre cells. The cells are polygonal in cross section and had a maximum diameter of up to 30 μm and a mean length of 1.1 (± 0.2) mm. Two cell-wall layers enclosed the lumen; the thin primary and the secondary cell wall, with a total thickness of ca. 2.1 (± 0.4) μm . Cross-sectional areas of the fibres differed significantly between 0.103 (± 0.06) mm^2 for samples from the plantation and 0.075 (± 0.04) mm^2 for those from the greenhouse. The microfibril angle (MFA) of the secondary cell wall of plantation and greenhouse samples accounted for 8° - 10° and 9° - 20° respectively. See Table 1.

3.2 Density, Contents of Cellulose, Hemicellulose and Lignin

The samples originated from the same plants that were used for mechanical tests. A total

amount of 3 g (greenhouse) and 6 g (plantation) fibres were used. The density of fibres differed between 0.84 (± 0.09) g/cm^3 (greenhouse) and up to 0.86 (± 0.07) g/cm^3 (plantation) (see Table 1). Furthermore, 10 g per plant was the minimum to determine the contents of cellulose, hemicellulose and lignin. Fibres from plantation and greenhouse exhibited nearly identical contents of lignin, cellulose and hemicellulose: 20.3%, 50.5% and 29.4% (greenhouse) as well as 20.3% 52.8% and 29.1% (plantation).

3.3 Mechanical Properties

In total 40 samples per source (plantation/Nicaragua and greenhouse/Germany) were subjected to tension tests and were used for calculating Young's moduli, tensile strength and breaking strain. On average, plants from the greenhouse had thinner fibre bundles than those from the plantation (0.08 (± 0.03) mm^2 versus 0.10 (± 0.05) mm^2). The same applies to Young's moduli. While fibres from plantation plants reached ca. 10.7 (± 3.7) GPa fibres from greenhouse plants showed ca. 4.4 (± 2.5) GPa. Tensile strength differed between 101 (± 56) MPa (plantation) and 49 (± 35) MPa (greenhouse). Breaking strain accounted for 1.4 (± 0.5)% (greenhouse) and 1.2 (± 0.3)% (plantation). See Table 1.

Table 1. Material properties of papaya fibres

	Plantation, Nicaragua	Greenhouse, Germany
Tensile strength [MPa]	101 (± 56)*	49 (± 35)*
Breaking strain [%]	1.2 (± 0.3)*	1.4 (± 0.5)*
Microfibril angle [deg]	8 - 10	9 - 20
Cross-sectional area [mm^2]	0.103 (± 0.06)*	0.075 (± 0.04)*
Density [g/cm^3]	0.86 (± 0.07)	0.84 (± 0.09)

Values are means and standard deviations are bracketed.

** denote significant differences ($P=0.05$) between results from plantation and greenhouse*

4. DISCUSSION

The obtained results serve as a first characterisation of the mechanical properties of papaya phloem fibres. A comparison of papaya fibres with those of other plant derived fibres shows, that Young's moduli of fibre samples from plantations (almost 11 GPa) are in the range of abacá (12 GPa), curauá (11.8 GPa) or bamboo (11 - 17 GPa) [2]. These values are distinctly lower than synthetic fibres or flax (27.6 - 54 GPa)

and hemp (17 - 70 GPa) fibres [2,18,19,20]. The tensile strength of papaya fibres (101 MPa) is rather low in comparison to other plant fibres. Similar low values of tensile strength could be found for bamboo (140 - 450 MPa) and coir (131 - 250 MPa) [2,21,22,23]. One reason for low breaking stresses can be short fibre cell lengths. Both bamboo and coir have fibre cell lengths of 2.9 mm and 0.8 mm respectively [24,25], compared to papaya fibre cells with ca 1.1 mm. Indeed, fibre cells with greater lengths; for example flax ca. 20 mm and hemp ca 23 mm [26] show higher breaking stresses [27,28]. Also values of breaking strain (1.2%) are lower than other natural fibres. The nearest value is 1.6% for hemp, jute and kenaf, respectively [2]. The Young's modulus of fibres from greenhouse plants (ca 4.4 GPa) is comparable to coir (4 - 6 GPa) and oil palm fibres (3.2 GPa) [2,22]. Tensile strength (49 MPa) is well below all other fibres while the breaking strain (1.4%) again corresponds to hemp, jute and kenaf [2].

Even though it seems that papaya fibres represent a comparatively weak material, we found that the density of papaya fibres is low with about 0.85 g/cm³. High Young's modulus in fibres is connected to a much higher density as shown for hemp and jute 1.3 - 1.5 g/cm³ (Table 2). Considering now the specific Young's modulus of these fibres we notice that papaya fibres range in the same sphere of lowest given values of specific modulus of high-modulus fibres. Because of this particular low density papaya fibres could be of interest for applications in green composites. Calculating a specific tensile strength, papaya fibres remain still below average (Table 2).

Comparing fibres with regard to the origin of our investigated plants shows that each of the mechanical properties, Young's modulus, tensile strength and elongation at break, varies significantly. The plants, however, grew under completely different environmental conditions regarding wind load, solar radiation, and day length or soil quality and volume. Especially due

to the lack of external loads such as wind in a greenhouse and the absence of fruits, the difference in mechanical properties is hardly surprising. However, the variation appears most likely with increasing age. The greenhouse plants had an age of six months, whereas the Nicaraguan plants were about 2 years old. One important criterion is the microfibril angle (MFA), which is known to change with the age of a plant – for trees. Higher MFAs lead to lower Young's moduli and larger breaking strains whereas lower MFAs have the opposite effect [29]. Fibres of young individuals which need to be flexible often have larger MFAs [30]. Older plants need to be stiffer to upright their organs and therefore possess fibre cells with smaller MFAs [30]. Our observations indicate that MFA of *C. papaya* adapts likewise, since Young's moduli increase with age of plants.

Lignin, cellulose and hemicellulose contents were almost identical when comparing the fibre origin. A high variability of mechanical properties can thus be a result of irregular cell geometry and fibre cross-sectional area [31]. Natural fibres have a lumen, which is usually not subtracted from the cross-sectional area of the fibre. Fibre diameter and lumen size are linked, so that the higher the fibre cross-sectional area, the higher the discrepancy in the calculation of Young's modulus and tensile strength. As a consequence the calculated Young's modulus and tensile strength is lower than the actual value would be. That is demonstrated by the fact that Young's modulus and cross-sectional area as well as tensile strength and cross-sectional area correlate significantly in our results (Table 3).

We investigated the potential of papaya fibres for use in green composites, fibre boards, etc. only from the view of mechanical properties of the fibres. Other attributes such as processing, response to moisture, thermal or insulating properties might be subject of further investigations. The compatibility of fibre and matrix material concerning the elastic and fracture behaviour is crucial for a composite.

Table 2. Comparison of hemp, jute and papaya fibers

	Hemp	Jute	Papaya
Tensile strength [GPa]	0.7 [22] - 1.7 [28]	0.4 - 0.8 [22]	0.05 - 0.1
Specific gravity	1.5 [22]	1.3 - 1.5 [22]	0.85
Specific Youngs modulus [GPa]	11 - 47	10 - 37	5.2 - 12.6
Specific tensile strength [GPa]	0.4 - 1.2	0.3 - 0.5	0.1 - 0.2

References are given in parentheses

Table 3. Spearman rank correlation of young's modulus *E*, tensile strength *TS* and fibre cross-sectional area *A*

Comparison	Origin	Spearman rank correlation	Significance
<i>E</i> – <i>A</i>	Greenhouse, Germany	-0.86	$3 * 10^{-15}$
<i>TS</i> – <i>A</i>	Greenhouse, Germany	-0.76	0.0006
<i>E</i> – <i>A</i>	Plantation, Nicaragua	-0.80	$3 * 10^{-10}$
<i>TS</i> – <i>A</i>	Plantation, Nicaragua	-0.57	0.01

Degrees of freedom: Germany 14, Nicaragua 17

In case of tension loads of the composite, the ratio of breaking strain of the fibres to that of the matrix is recommended to be 1:3 at least. Since tension forces are taken up mainly by fibres, the respective Young's moduli should be larger than those of the matrix. For a first estimation considering fibres derived from plantation plants, Young's modulus of the matrix should be smaller than 11 GPa and breaking strain should be larger than 3.6%. For example vinyl ester resin (Young's modulus = 3.4 MPa, breaking strain = 3.5 - 7.0%) or epoxy resin (Young's modulus = 2.8 - 3.6 MPa, breaking strain = 6 - 8%) would represent suitable matrix materials [32].

Characteristic values such as Young's modulus, tensile strength and breaking strain exhibit a high variability of materials properties. However, biological materials characteristically show a broad variation in acquired properties with regard to location-dependent conditions, such as temperature, wind exposure or nutrient supply, as well as age of used plants. It should be the aim of further research to verify the impact of these factors. Plantations are renewed every 3 - 5 years for economic reasons, so that age of plants already is limited. Reasonable applications for these composites might be components subject to low mechanical stresses, e.g. interior parts in automotive industries (panels in car doors) or in package industries (housings). The use of entire fibre mats as design elements emphasizing its peculiar fibre arrangement (see Fig. 1B) is conceivable, too. Since papaya plants were cultivated for fruits fibre material accumulates in a huge amount on plantations, which is still unused. A rather conservative estimate reveals 1.2 million tons of fibre material in 3 - 5 years. A current area of commercial plantations summing up to about 406,000 ha in 2011 [10] is taken as basis, calculated with one plant per 10 m² (estimated) and ca. 3 kg fibres per stem estimated from 5% fibre content per stem [9]. Although not all of the accruing fibres are usable, the remaining material would represent a substantial amount bearing in mind that the world production of jute, sisal, and flax

are 3.6, 0.4, and 0.2 million tons, respectively, in 2011 [10].

5. CONCLUSION

A high variability existed in the investigated material properties Young's modulus, tensile strength and breaking strain. Plant age, growth conditions and the mode of fibre extraction most likely account for that. Compared to mechanical properties of other natural fibres Young's modulus, tensile strength and breaking strain are average. Otherwise papaya fibres exhibit noteworthy specific Young's modulus due to low fibre density. Fibre material accumulates in a huge amount on plantations, which could be an easy accessible and cheap source for further utilisation. Based on investigation of material properties papaya fibres provide a new opportunity of application in the field of green composites. For this purpose pursuing research is necessary dealing with fabrication and testing of first composite prototypes made of papaya fibres.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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