DIMENSIONAL VARIATIONS AND MECHANICAL BEHAVIOUR OF NATURAL FIBRES FROM VARIOUS PLANT SPECIES IN CONTROLLED HYGRO/HYDROTHERMAL CONDITIONS

William GARAT[1,2], Stephane CORN[1*], Nicolas LE MOIGNE[1*], Johnny BEAUGRAND[2,a] and Anne BERGERET[1]

[1]C2MA, IMT Mines Ales, Université de Montpellier, 6 avenue de Clavières, 30100 Alès, France
[2] Fractionnement des Agro Ressources en Environnement (FARE), INRA, Université de Reims Champagne Ardenne, 2 esplanade Roland Garros, 51100 Reims, France

*Email: stephane.corn@mines-ales.fr, nicolas.le-moigne@mines-ales.fr

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Abstract

Natural fibres are considered as ecological alternative reinforcements to glass fibres in polymer composites. However, the behaviour of biocomposites in service life and their durability are impacted by the great variability in dimensions and mechanical properties of plant fibres. Beyond the fact that each plant species has its own intrinsic properties, these variations can be explained in particular by a high sensitivity of plant fibres physical properties to hygro/hydrothermal conditions. In order to better understand and quantify these phenomena, we studied the effect of environmental conditions on the dimensional variations and the mechanical behaviour of fibre bundles extracted from several plant species from different botanical origins. All tests were conducted in both a climatic chamber or water immerged allowing to vary the relative humidity conditions between 20 to 100%. Our results revealed contrasted swelling and plasticization behaviour as a function of plant species, in relation with their specific biochemical composition and microstructure, influencing greatly their mechanical properties. This study highlights the need to characterize natural fibres under controlled hygro/hydrothermal conditions. In addition, the analysis of their swelling and mechanical properties as a function of RH opens interesting perspectives to better understand and estimate mechanical behaviour of biocomposites in service life.

1. Introduction

Currently, industry is showing a growing interest in the development of biocomposites. Natural fibres of various botanic origins are used as reinforcements in polymer composites [1–3] and can be considered as a solution to current environmental issues, including the reduction of greenhouse gas emissions and pollution, through the use of renewable and biodegradable resources and the development of lightweight materials. Up to now, natural fibres lack scientific and technological backgrounds to claim a controlled and constant level of performances, which drives agricultural and industrial sectors to collaborate to reach end-users expectations. In order to be able to control, model and accurately predict the mechanical behaviour of biocomposites during their processing and service use, it is essential to have a thorough knowledge of the morphological characteristics and mechanical properties of natural fibres especially because they highly depend on humidity and temperature conditions [4–8]. Some authors proposed elements that are useful to address these questions. Pejic et al. [9] investigated the role of non-cellulosic biopolymers as hemicelluloses and lignin on the swelling

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[a] Current address : Biopolymères Intractions Assemblages (BIA), INRA,
Rue de la Géraudière, 44316 Nantes, France Johnny.beaugrand@inra.fr

phenomenon of hemp fibres. They studied the water absorption behaviour of hemp elementary fibres and fibre bundles for different biochemical compositions after extraction by chemical treatments. Results showed that when the content of non-cellulosic biopolymers was progressively reduced, the water uptake of hemp elementary fibres consequently increased up to 2.7 times and even more emphasized for fibre bundles. Other authors studied the effects of environmental conditions on the tensile stiffness and strength of flax and nettle elementary fibres. Davies et al. [6] observed that the average decrease rates of the tensile modulus were 0.39 GPa per %RH for flax fibres and 0.24 GPa per %RH for nettle fibres over the range 30-70% RH. More recently, Placet et al. [10] showed that water sorption has a significant influence on the tensile stiffness and strength of hemp fibres under cyclic loading. They observed an unexpected increase of the E-modulus and tensile strength up to 70% RH, followed by a decrease at higher RH, which was related to the plasticization of amorphous polysaccharides that occurs beyond this water saturation point (70% RH), leading to a loss of mechanical properties.

The purpose of this work is to characterize the dimensional variations and the mechanical behaviour of natural fibre bundles extracted from various plant species in various hygro/hydrothermal conditions. Based on an automated laser measurement technique of the cross-sectional area [11] along the fibres and a micro-tensile test, both conducted in a climatic chamber or immersion cell, the variations in transverse dimensions and the mechanical behaviour of natural fibre bundles have been studied.

2. Experimental section

2.1. Materials

Characterizations of dimensional variations and mechanical behaviours of natural fibres were conducted for fibre bundles extracted from five various phylogenetic plant species with dissimilar functions *in planta*. Due to their various botanical origins, these fibre bundles exhibit contrasted dimensional and morphometric characteristics [11]. Three of them belong to eudicots and are commonly called ‘bast fibre’, poorly lignified (<5%), with a support function of the stem *in planta*: flax (*Linum usitatissimum*) and hemp (*Canabis sativa*) as annual fibre crop (after retting step) and nettle (*Urtica dioica*) as an perennial herbaceous plant. The other two species are from perennial monocots, highly lignified (>9%), with a dominant conducting tissues function in planta: sisal (*Agave sisalana*) coming from the leaf of agave and palm (*Phoenix dactylifera*) are mats of the leaf sheath surrounding palm tree stems.

2.2. Characterization methods of natural fibre bundles

2.2.1. Dimensional variations analysis

A Fibre Dimensional Analysis System (FDAS) device (Diastron Ltd, Hampshire, UK) was used to measure the fibre bundles transverse dimensions and thus quantify the cross-sectional area (CSA) longitudinal variations. It is an automated experimental method based on laser scanning described in details in our previous study [11]. For each species, the non-circularity of fibre bundle cross-sections and their variability along the fibre length were taken into account. A filtered elliptic model using the maximum and minimum apparent diameters of each of 42 cross-sections per fibre bundle was applied for the calculation of its CSA.

2.2.2. Mechanical behaviour analysis

Mechanical properties, i.e tensile strength and Young’s modulus, have been obtained from micro-tensile tests, using a Linear Extensometer (LEX) device equipped with a 20N capacity load cell and controlled by UvWin 3.60® software (Diastron Ltd, Hampshire, UK). Force to stress conversion was based on the aforementioned CSA calculation. Tests were carried out at a displacement speed of 0.02 mm/s and a gauge length of 30 mm was choosen. The Young's modulus and the failure strength have been determined in the linear zone near failure (between 1 to 3% strain in this study) according to the NF T25-501-3 standard.

2.2.3. Control of hygro/hydrothermal conditions

The objective of these *in-situ* tests is to analyse the influence of relative humidity on the transverse dimensions and the mechanical behaviour of fibre bundles. Both analysis apparatus (dimensional (FDAS) and mechanical (LEX)) were placed in a climatic chamber (Fig. 1) supplied by ETS company (Electro-Tech Systems Inc, Pennsylvania, USA) to regulate temperature at 23°C and relative humidity at 20, 50 and 73% RH). At least 20 fibre bundles of each plant species were tested for each relative humidity conditions after being acclimatized for more than 13 h.

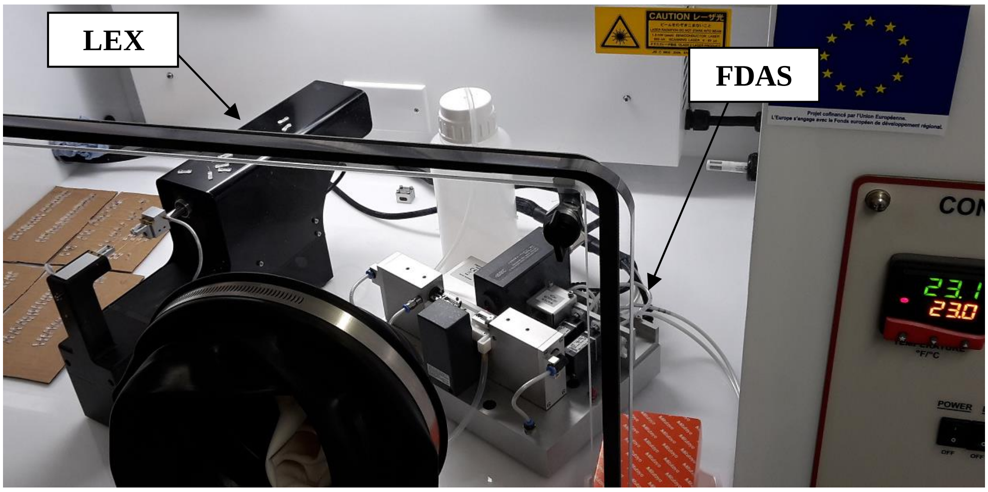

Figure 1. Fibre Dimensional Analysis System (FDAS) device and Linear Extensometer (LEX) device positioned in the controlled climatic chamber.

A dedicated immersion cell (DSM cell, Diastron Ltd., Hampshire, UK) was implemented on the FDAS apparatus for measuring their dimensions in controlled hydrothermal condition, i.e. 100% RH. Fibre bundles were immersed in deionized water at 23°C for dimensional measurements (FDAS) and directly positionned on the LEX apparatus for tensile testing. At least 20 fibre bundles of each plant species were tested after being acclimatized for more than 13 h at 23 °C and 50% RH.

3. Results and discussions

3.1. Influence of relative humidity on dimensional variations

3.1.1. Variation of the fibre cross-sectional area

The influence of the relative humidity on the dimensional variations of natural fibre bundles and especially on the swelling of their cross-section was analysed. The evolution of their relative median CSA (normalized to their CSA at 50% RH) was measured at every relative humidity (Fig. 2).

At 20 %RH, the shrinkage of the fibres is low, i.e. 3 ± 1 %, althought being more pronounced in the case of nettle fibre bundles, i.e. 7 ± 2 %. At 73 % RH, the swelling is low for all plant fibre species (inferior to 10%) with little dispersion. In immersion conditions, the swelling drastically increases. Large differences are observed depending of plant fibre species with swelling magnitudes varying from 19.5 ± 6 % for palm fibres and up to 74 ± 15 % for nettle fibre bundles. It is worth to point out that highly lignified fibre bundles as palm and sisal tend to have a lower cross-section swelling than weakly lignified fibre bundles as flax, hemp and nettle (Table 1). The influence of non-cellulosic biopolymers as lignin on water absorption and swelling of hemp fibres has been studied by Pejic et al. [9]. According to the authors, lignin removal increased the water retention ability of hemp fibres.

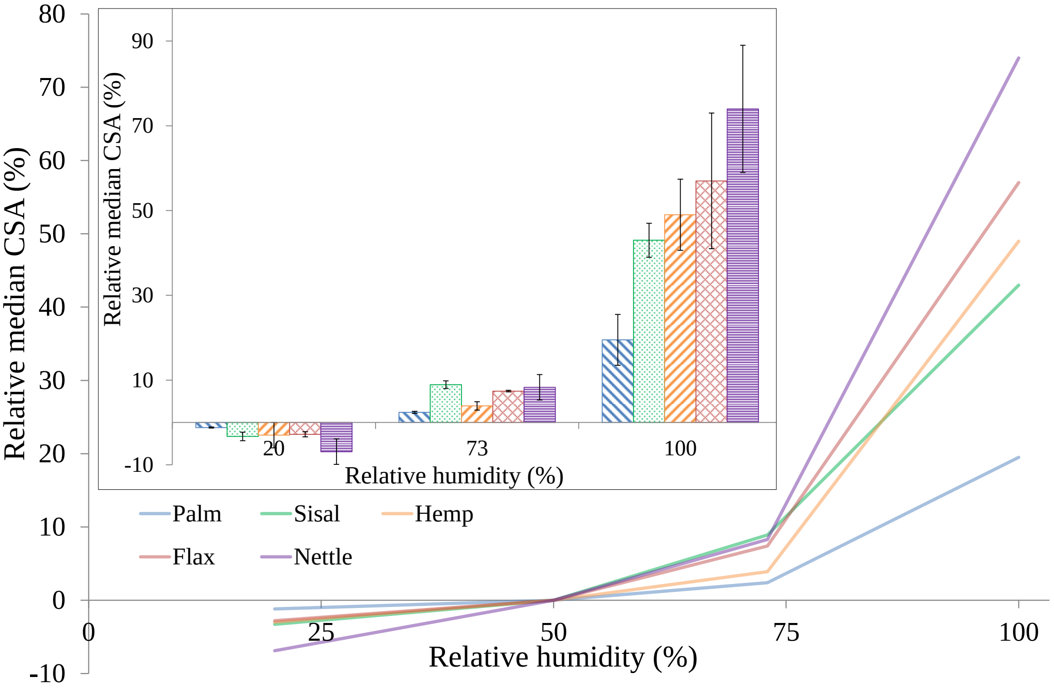

Figure 2. Variations of relative median CSA normalized to their CSA at 50% RH as a function of the relative humidity for different plant fibre species.

3.1.2. Variation of the fibre cross-sections shape factor

Fig. 3 presents the evolution of the median shape factor (α), i.e. the ratio of the maximum to the minimum diameters of fibre bundle cross-section, as a function of relative humidity as measured for the different plant fibre species [11]. The shape factor is a good morphometric indicator of the non-circularity of fibre cross-section. The relative humidity has few influence on palm and sisal fibre bundles with cross-sectional shape factors close to 1, i.e. circular shape. Indeed, their median shape factors remain similar, i.e. 1.20 ± 0.07 and 1.27 ± 0.11 for palm and sisal respectively, whatever the relative humidity from 20 to 100% RH. It can thus be concluded that the swelling of their cross-sectional is radially homogeneous. In contrast, hemp, flax and nettle fibre bundles present higher median shape factors with wider dispersion which is influenced by the relative humidity especially at 100% RH.

The increase of their minimum diameter is greater than the one of their maximum diameter leading to significant decrease of their shape factor in immersion. Hemp, flax and nettle fibre bundles present median shape factors of 1.81 ± 0.26, 1.92 ± 0.42 and 1.97 ± 0.69 respectively, at 100 % RH which makes these fibre bundles less flattened during immersion in water (Table 1). It can be assumed that this heterogenous swelling effect is due to the rehydration of elementary fibres that were flattened when the cells desiccated at the end of growth. The lower the thickness of the secondary cell walls, the higher the flattening effect upon desiccation and the higher the decrease of the shape factor upon rehydration.

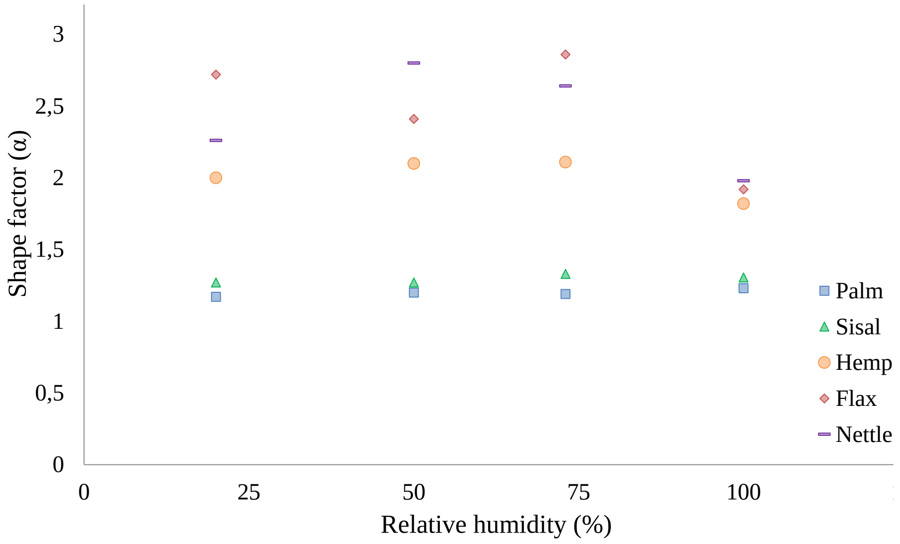

Figure 3. Evolution of the median shape factor as a function of relative humidity for different plant species

Table 1. Dimensional characteristics and variations of natural fibre bundles at 50 and 100% RH

Fibre bundles	Relative humidity (%)	Shape factor (α)	Relative median CSA / CSA at 50 % RH (%)	Median increase (%) Maximum diameter	Median increase (%) Minimum diameter
Palm	50	1.20 ± 0.07	-	-	-
	100	1.23 ± 0.06	19.5 ± 6	9 ± 3.7	10 ± 1.8
Sisal	50	1.27 ± 0.11	-	-	-
	100	1.31 ± 0.12	42 ± 3	14 ± 4	23 ± 5
Hemp	50	2.10 ± 0.35	-	-	-
	100	1.81 ± 0.26	49 ± 8	15 ± 2.5	27 ± 6.8
Flax	50	2.41 ± 0.42	-	-	-
	100	1.92 ± 0.42	57 ± 15	17 ± 5	35 ± 8
Nettle	50	2.80 ± 0.55	-	-	-
	100	1.97 ± 0.69	74 ± 15	18 ± 5.5	54 ± 16

3.2. Influence of relative humidity on the mechanical behaviour

In general, palm and sisal fibre bundles have lower mechanical properties than bast fibres. Flax fibre bundles present best properties regardless the tested relative humidity. Fig. 4 shows the effect of relative humidity on the mechanical properties of natural fibre bundles. An increase in tensile strength for bast fibre bundles (hemp, nettle and flax) with increasing RH up to a threshold RH value has been observed. Beyond this threshold RH value in the vicinity of the water saturation point, the plasticization of amorphous polysaccharides within the cell walls occurs [4,10] leading to a drop of the tensile strength as for exemple for flax fibre bundles with a decrease of 245 MPa from 73 % RH to 100 % RH. The tensile strength of palm fibre bundles decreases of 52 MPa (corresponding to a decrease of 47%) between 20 to 73 % RH with no additional decrease in immersion. It should be pointed out that a variable threshold value comprised between 50 and 73% RH is clearly distinguished for the different plant fibre species and has to be related to variation in their water saturation point. Concerning the Young's modulus, it decreases when the RH is increased for all the plant fibre species as for exemple for flax fibre bundles with a decrease of 15 GPa from 20 to 100 % RH. Only for nettle fibre bundles, the Young's modulus increases of 7 GPa between 20 to 50 % RH before decreasing in immersion. These results evidence that water molecules contribute to plasticize the cell walls and decrease the stiffness of fibre bundles whatever the plant species considered [6,8,9].

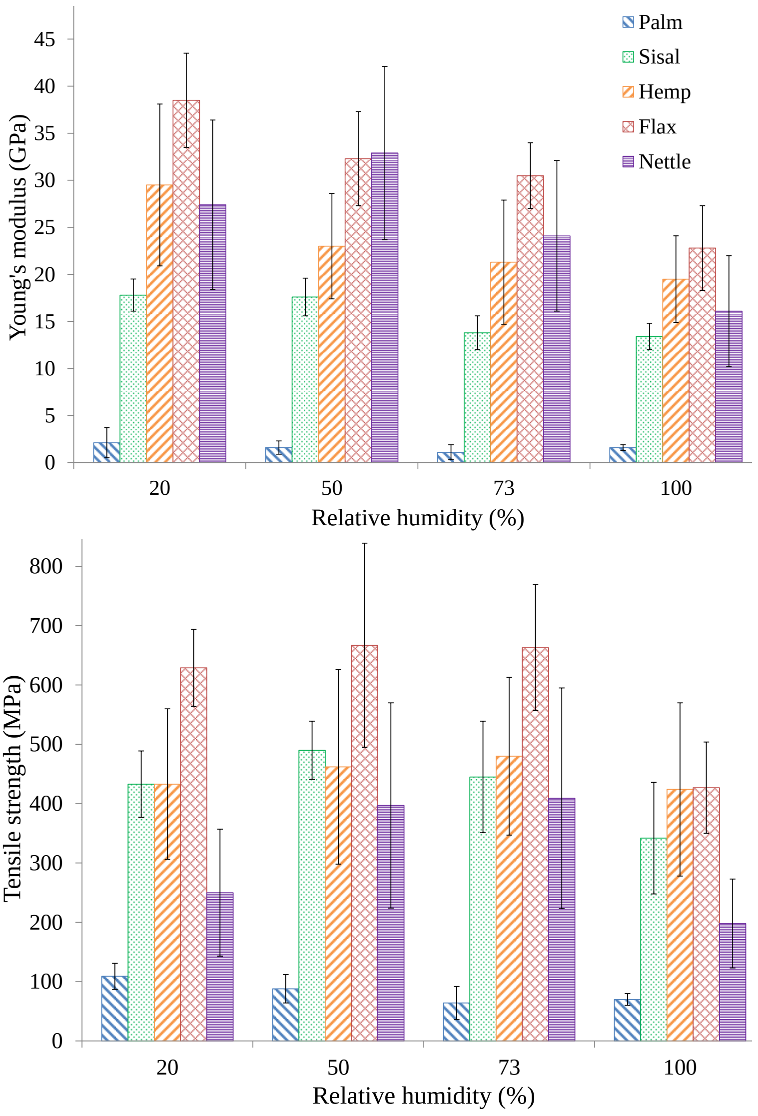

Figure 4. Influence of the relative humidity on mechanical properties (a) tensile strength, (b) Young's modulus of bundles from different plant fibre species.

A probability distribution (determined for a probability of failure of 50%) was used to analyze the failure probability distribution of fibre bundles. Several authors have already used this Weibull statistical approach, as Silva et al. [12] for sisal fibres and Placet et al. [10] for hemp fibres. The Weibull modulus describes the degree of scattering of failure strength, which can be related to the heterogeneity of the fibre bundles microstructure. It allows to determine a characteristic tensile strength of the different plant fibre bundles for each RH condition. Values of the Weibull modulus are relatively high compare to values in literature (as for exemple 1.5 for flax fibre bundles), which reflects the homogeneity in the distribution of the defects size for the tested fibre bundles, characteristic of a low dispersion (Table 2). However, the relative humidity has a significant influence on the values of the Weibull modulus suggesting that the dispersion of the tensile strength values is RH dependent. Caracteristic tensile strength values are relatively close to median tensile strength values, for exemple at 50% RH, the Weibull prediction for flax fibre bundles is 673 MPa compared to the median tensile strength of 667 ± 172 MPa. These results highlight that the Weibull statistic is an interesting approach to analyse the failure behaviour of fibre bundles as a function of relative humidity conditions.

Table 2. Characteristic tensile strength σ_0 and Weibull modulus as a function of RH for different plant fibre species.

Fibre bundles	**Weibull parameters**	**Relative humidity (%)**			
		20 %	**50 %**	**73 %**	**100 %**
Palm	**Characteristic strength σ_0 (MPa)**	110	88	61	71
	Weibull modulus / R^2	4.25 / 0.98	3.43 / 0.96	2.23 / 0.78	5.51 / 0.97
Sisal	**Characteristic strength σ_0 (MPa)**	439	497	450	335
	Weibull modulus / R^2	7.6 / 0.94	9.1 / 0.96	4.4 / 0.94	1.8 / 0.82
Hemp	**Characteristic stress σ_0 (MPa)**	424	455	477	408
	Weibull modulus / R^2	2.5 / 0.95	2.8 / 0.92	2.8 / 0.96	2.2 / 0.92
Flax	**Characteristic stress σ_0 (MPa)**	638	673	671	432
	Weibull modulus / R^2	8.21 / 0.94	3.68 / 0.97	5.69 / 0.94	5.16 / 0.96
Nettle	**Characteristic stress σ_0 (MPa)**	240	391	383	193
	Weibull modulus / R^2	2.1 / 0.97	1.8 / 0.95	2.0 / 0.89	2.6 / 0.90

4. Conclusions

Our results have shown that relative humidity has significant influence on the transverse dimensions and mechanical behaviour of fibre bundles, their botanical origin being an important influential factor. It was found that swelling in controlled RH conditions differs between plant fibre species due to variations in their biochemical composition and microstructure. In particular, it was found that weakly lignified fibre bundles as flax, hemp and nettle showed much higher swelling in immersion. Interestingly, their cross-section shape factor is also greatly affected and drastically decreased in immersion, fibre bundles being thus less flattened when in high RH conditions. Concerning the mechanical behaviour, relative humidity has a negative influence on the Young’s modulus which decreases with increasing RH whatever the plant species considered, highlighting a strong plasticization effect of the cell walls. Besides, tensile strength increase with increasing RH up to a threshold RH value before sharply decreasing at higher RH. This variable threshold value, which was comprised between 50 and 73% RH, has to be related to variations of the water saturation point for the different plant fibre species. Moreover, our work points out that the Weibull statistic is an interesting approach to analyse the influence of the relative humidity on the fibre bundles strength, given the dispersion of the results often reported in literature.

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