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Abstract
Over the past 20 years, a large number of studies have been carried out to determine the mechanical properties of plant fibres. Most studies focus on the use of tensile tests on elementary fibres. Despite its advantages, this method is also time-consuming and challenging because of the tiny size of the fibres and the many sources of uncertainty.
The aim of the present benchmark study was to gain a better understanding and quantify the sources of variability when estimating the tensile properties of plant fibres. For that purpose, three batches of fibres were selected: two plant fibres, i.e. combed hemp and flax fibres as well as a synthetic organic fibre, i.e. aramid fibres, for benchmark purpose. A total of approximately 1250 fibres were tested. 9 research groups participated in this interlaboratory exercise.
Results highlight significant intra- and inter-laboratory variability. Human factors and experimental procedures, especially for the estimation of the fibre cross-sectional area and the tensile strain, were identified as the main sources of scatter in tensile properties. Post-processing procedures, particularly determining the starting point of the tensile test, are also crucial, involving the elimination of slacks in fibre and load trains.

1. Introduction
Three types of tests are commonly used to determine the tensile properties of plant fibers [1]: tensile tests on single individual fibers [2], tensile tests on bundles of fibers [3], and the inverse method IFBT (Impregnated Fiber Bundle Testing) [4]. A large number of studies focuses on the use of tensile tests on elementary fibres. This method is complex and time-consuming due to the small dimensions of the fibres. Several standards, such as ASTM C1557 [5], ASTM D3822 [6], and NF T25-501-2 [7], have been specifically developed for conducting tensile tests on single fibres. ASTM C1557 focuses on determining the tensile strength and Young's modulus of advanced ceramic, glass, carbon, and other fibres. ASTM D3822 is tailored for assessing the tensile properties of both natural and synthetic fibres. Additionally, NFT25-501-2 is intended for determining the tensile properties of single flax fibres. These standards provide guidelines for carrying out tensile testing procedures. However, amendments are necessary to better account for the specificities of plant fibres compared to synthetic fibres and monofilaments in the experimental methods and protocols, and to minimize epistemic uncertainties.
To advance in this direction, a work group (WG) has been established within the framework of the GDR ("Groupement de Recherche") MECAFIB (Mechanics of fibrous materials). GDRs are native entities of CNRS, the French Nation Centre for Scientific Research, that bring together and federate a scientific community around an emerging, original research topic. This WG formed in this context consists of nine research groups, all experienced in conducting tests on plant and/or synthetic fibres. An interlaboratory exercise was organized within this group to better assess the variability of the tensile properties of plant fibre properties, to identify and rank more effectively the main sources of epistemic uncertainties, and ultimately produce recommendations to ensure that dispersion values resulting from single fibre test predominantly reflects the intrinsic variability of fibre properties.
Tests were conducted on two plant fibres, hemp and flax, alongside aramid for benchmarking puposes, as this synthetic fibre shares dimensions and stiffness properties similar to the selected plant fibres. Flax and hemp fibres were chosen due to their prevalence among European fibre plants and extensive use in both textile and composite industries.

2. Materials and methods

2.1. Fibres
The flax fibres used in this interlaboratory exercise were sourced from Bolchoï variety plants grown in Normandy, France, in 2018. The hemp fibres were extracted from Futura 75 variety plants cultivated in Piacenza, Italy, also in 2018. The tested fibres were obtained after scutching and hackling. For aramid, the high-performance K29 para-aramid fibre produced by Dupont ™ was used. Fibres from each type were distributed to all nine laboratories participating in this benchmark exercise. The laboratories are designated by letters ranging from A to K.

2.2. Experimental methods

Individual elementary fibres were used for the single fibre tensile tests. The fibre selection, individualisation into single fibres, preparation, morphological characterisation and tensile testing were carried out following the typical procedures, methods, and equipment used in the different participating laboratories. For flax and hemp, fibres with apparent diameters exceeding 30 µm and 40 µm, respectively, were excluded from the analysis due to the high likelihood of comprising multiple elementary fibers.

Upon initiating the benchmark study, each participating laboratory received a specific protocol outlining recommendations for fibre preparation and conditioning, mounting conditions (paper cardboard), gauge length (10 or 12 mm), displacement rate (1 mm/min) during tensile testing, the number of fibres to be tested, data acquisition conditions (force and displacement/strain, relative humidity, temperature), and data processing. However, due to disparities in available equipment, some adaptations were necessary in certain cases.

The initial apparent tangent modulus (E_i) and the final apparent tangent modulus (E_f) were determined using two different strain ranges (between 0.05% and 0.5 % for E_i and between ε_{max}-0.2% and ε_{max} for E_f). The stress and strain at failure (σ_R and ε_R) were determined by looking for a 30% drop in stress. The preceding data point before this drop delineates the stress and strain at failure.

Statistical tests were used to assess whether the determined tensile properties for each fibre type across the different laboratories significantly differ from each other.

3. Results

Figure 1 synthetizes the tensile properties identied for the three fibre types by the nine research groups. For aramid fibres, mean values of initial apparent tangent tensile moduli E_i, tensile strength σ_R, and strain at failure ε_R fell within the ranges of 67-97 GPa, 2821-3981 MPa and 3.41% to 4.5%, respectively. This is in good agreement with data previously reported in literature for K29 fibres (78.1 ±9.6 GPa, 3 300 ±500 MPa and 3.8 ±0.3%) [8]. For flax, the values were in the ranges of 11.4-52.8 GPa, 410-1130 MPa, and 1.2% to 2.91%. For hemp, the ranges were 18.5-34.7 GPa, 322-699 MPa, and 1.47% to 3.1%. The values obtained in this study are therefore on the lower end of the published data for flax and in the mid-range for hemp [9]. One of the main sources of the inter-laboratory variability of the apparent initial modulus originates from the non-linearity of the responses of most of the tested plant fibres and the variability of the non-linear patterns observed. One of the main sources of variability in the overall tensile properties measured is also the determination of the effective starting point of the tensile test. Indeed, all tested fibres exhibit initial geometrical defects (such as curvature, twisting, etc.). Therefore, it is necessary to apply a pre-tension to align the fibre with the tensile axis and to eliminate any slack in the load train. At this small scale, achieving and measuring such a low pre-tension may be challenging, and not all commercial tensile testing machines necessarily provide access to this parameter/quantity. In the work presented, a unique post-processing procedure was applied to all datasets to reduce the uncertainty associated with this point.

Concerning, the intra-laboratory variability that for E_i and σ_R, it mainly results from uncertainties in the cross-sectional area measurements. It is markedly lower for aramid fibers, with coefficients of variation (CoV) ranging from 8% to 22% for Ei and 10-23% for σR, compared to flax fibers (CoV ranging from 28% to 54% and 32% to 59%, respectively) and hemp fibers (CoV ranging from 38% to-68% and 51% to 80%, respectively). The distribution of strength to failure also correlates with flaw sensitivity. These flaws may be inherent to the fibres themselves, originating from their production and processing, or introduced during fibre preparation and handling for testing. Additionally, multiaxial stress concentrations near the fibre ends within the gauge section, resulting from clamping, misalignment, or geometrical variations, can also contribute to premature failure.

For the strain at failure, the CoV ranged from 8 to 23% for for aramid fibres 23-44% for flax and 36-58% for hemp. The variability in this quantity is linked to both the method used to measure the global displacement of the fibre (from which the strain is derived) as well as the correction of the compliance of the testing system.

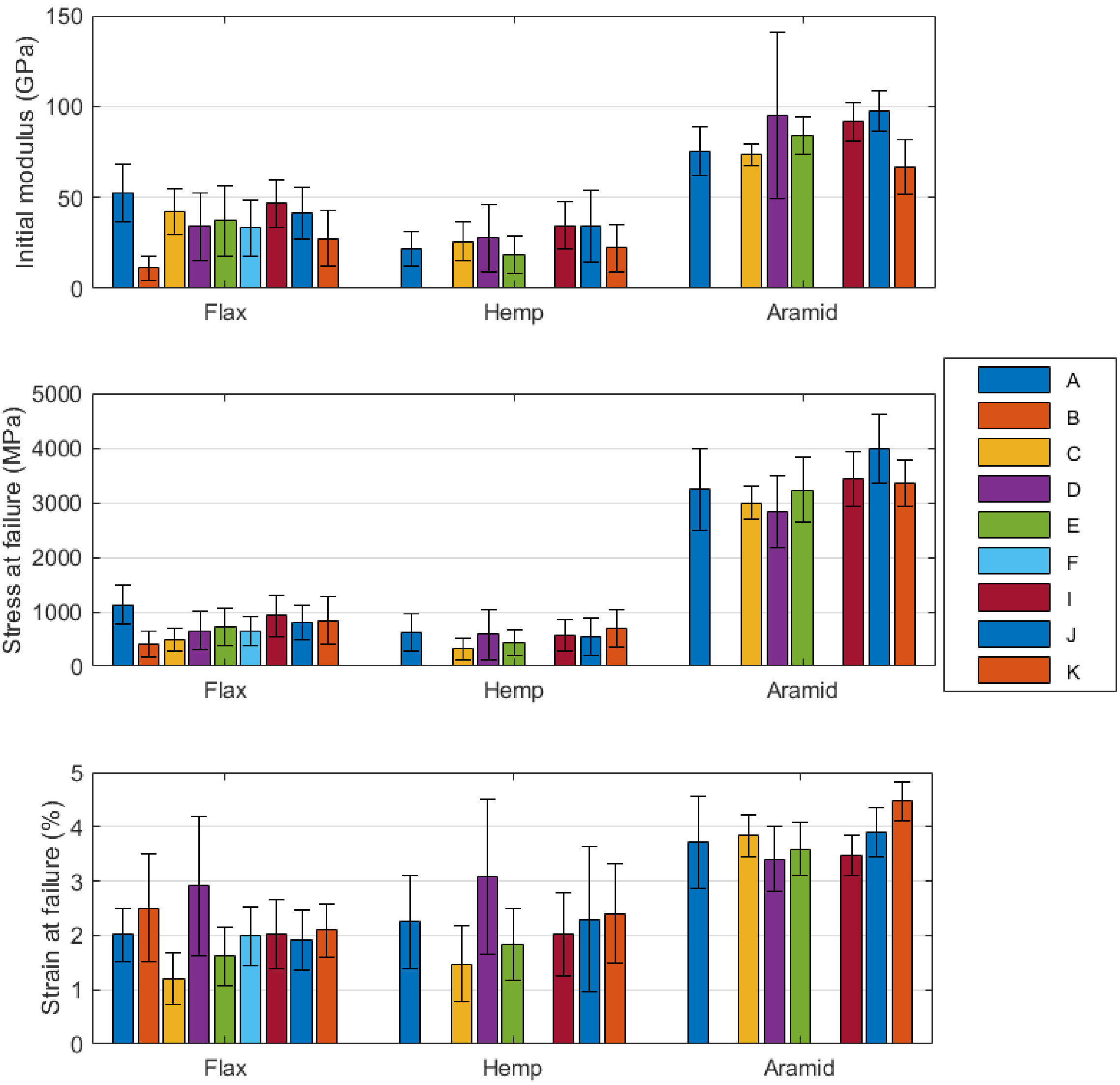

Figure 1. Tensile properties (E_i, σ_R and ε_R) determined by the different laboratories (A-K) for the three tested fibres types (flax, hemp and aramid). *Bar: mean values, error bars: ±standard deviation.*

4. Conclusions

Results highlighted significant intra- and inter-laboratory variabilities. The more pronounced scatter for flax and hemp compared to aramid was attributed to the difficulty to analyse the high variability and complexity of plant fibre cross-section and the greater diversity of non-linearities observed on stress-strain curves.

The main recommendations for reducing dispersion in tensile testing include considering geometric models adapted to fibre morphology when determining the cross-sectional area, maintaining precisely controlled hygrothermal conditions, accurately defining zero load and zero displacement points, and systematically specifying the strain range used to determine apparent tangent modulus. Finally, alternative methods such as Digital Image Correlation should be considered to reduce uncertainties in strain measurements.

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