MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HEMP TECHNICAL FIBRES FOR COMPOSITE APPLICATIONS BY MICRO COMPUTED TOMOGRAPHY AND DIGITAL IMAGE CORRELATION

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Abstract

This research presents a novel experimental approach to characterize the non-linear behaviour of hemp fibres, by combining standard tensile tests with a detailed strain analysis during tensile loading. First, the strength and Young's modulus distribution of technical hemp fibres of 78 different variants (fibre species and geographic location) were analysed using the modified Weibull distribution (length-based model), and one-way ANOVA statistical analysis. Next, strain mapping via digital image correlation analysis was used for evaluating the local mechanical behaviour heterogeneity along a technical natural fibre. The different tests performed allow a complete characterization of hemp fibres, which reveals 3 typical types of tensile stress-strain curves depending on their strength, and a complex and very irregular pattern of strain concentrations. Micro tomography is used to study the fibre microstructure and linked with the mechanical behavior analysis, in order to obtain a deeper understanding of the hemp fibre tensile behavior.

1. Introduction

The mechanical behaviour of polymer composites depends on the properties of the reinforcing fibre, the matrix and the fibre-matrix interface. However, natural fibres are composite materials themselves showing a hierarchical organization at different sub levels, and the mechanisms that govern their mechanical behaviour (hence the behaviour of their composites) are not completely understood.

The structure of a natural plant fibre consists of abundant cells (elementary fibres) with thick cell walls. The cells are surrounded by a soft amorphous lignin or pectin layer (middle lamella). The cell wall is, in turn, composed of several layers of rigid cellulose nanofibrils oriented at different angles, and joined by hemicelluloses and lignin as matrix material. Finally, nanofibrils are composed of an array of individual cellulose chains [1].

Due to this complex hierarchical structure, most natural fibres present a non-linear tensile behaviour, which might be the consequence of different micromechanical mechanisms that are occurring at different length scales. Standard methods for measuring tensile properties give global strain. It was evaluated if digital image correlation (strain mapping) could be used for the determination of strain heterogeneity along a technical natural fibre.

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The objective of this research is to study the non-linear tensile behavior of hemp fibres of 78 different variants (the factors included in the experimental design were the fibre species, and the geographic location where the plants were cultivated) and to explain the responsible mechanisms of their performance, as a part of the FP7 - Multihemp project (Multipurpose hemp for industrial bioproducts and biomass). The strength distribution of technical hemp fibres of the different variants is analyzed using the Weibull distribution and one-way ANOVA statistical analysis.

For further investigation, the mechanical behavior at two different levels (elementary and technical fibre) was then studied by using micro strain mapping. In order to consider a possible link between the microstructure and the tensile behavior, the fibre microstructure was studied by micro-tomography. Micro-computed tomography makes it possible to measure three dimensional geometries at submicron resolution.

2. Materials and Methods

2.1. Materials

26 hemp fibre varieties were provided by partners of the Multihemp project. Each variety was cultivated on three different geographical locations (The Netherlands, Italy, and France), making a total of 78 variants. The same fibre extraction method (decortication) was used for all the samples.

2.2. Tensile test

20 randomly selected technical fibres for each variant were visually inspected to verify the absence of major damage, and then conditioned at 20° C and 50% RH. The fibres were then weighed and the cross-sectional area of each individual fibre was calculated using its density, weight and length. The density of hemp fibres (1.56 g/cm^3) was measured in a gas pycnometer, and only fibres with cross–sectional areas within 0.005 and 0.025 mm^2 were used. A span length of 20 mm was used for all the tests.

A normality analysis is performed on the results with a confidence interval of 95%. All results out of this range are excluded, guarantying that no less than 15 fibres are finally used for Weibull analysis. The Weibull distribution is a statistical tool for describing the tensile strength of brittle materials under the assumption that fibre failure occurs when its weakest element fails.

Finally, a single factor analysis of variance (ANOVA) was used for verifying the difference in the mean strength between different variants.

2.3. Strain mapping

A random speckle pattern is created on fibers by using an Airbrush with a spray nozzle of 0.15mm.

The main parameters for a suitable speckle pattern for digital image correlation (DIC) analysis consist of speckle size, randomness and distribution. A good pattern, as illustrated in Figure 1, consists of evenly distributed and uniform speckles on the whole area to be analyzed.

In order to evaluate if the speckle patterns were good enough for DIC analysis, two methods were implemented: pixel intensity histogram and strain deviation analysis [2, 3].

An acceptable speckle pattern will produce a Gaussian distribution when it is subjected to a pixel intensity histogram analysis (see Figure 2 - Left).

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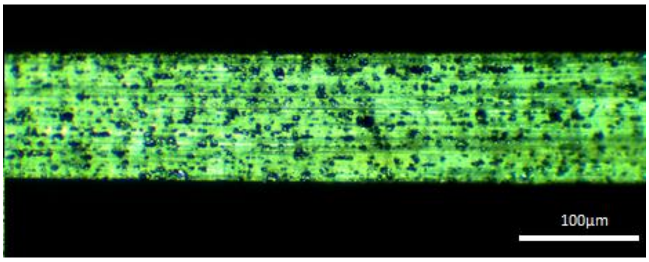

Figure 1. Example of a good speckle pattern on a hemp fiber.

Regarding the strain deviation analysis, a virtual and uniform deformation is applied on the image of the speckle pattern. Since the magnitude of the deformation is controlled and uniform, a homogeneous strain mapping over the entire image is expected. However, in reality digital resizing is not totally uniform, and local variations could be created depending on the method used for interpolation. Then, the quality of a speckle pattern could be evaluated by comparing strain deviations along a horizontal arbitrary chosen straight line on image length. A strain deviation analysis of a relatively good and relatively bad speckle pattern can be seen in Figure 2 – Right.

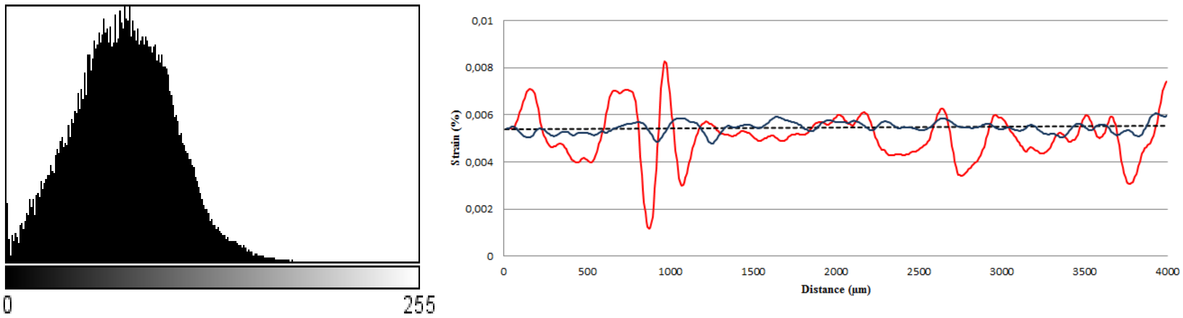

Figure 2. Pixel intensity histogram for the speckle pattern presented in Figure 1 (Left), and comparison of the strain deviation of a good (blue) and poor (red) pattern along the sample length (right).

A Motic microscope SMZ-171-TH with a Moticam camera of 10 MP of resolution were used to acquire the images. Ncorr software was used for DIC analysis.

2.4. Tomography

A SkyScan micro computed tomography (µCT) device for scanning electron microscope (SEM), and a Phoenix NanoTom X-ray computed tomography equipment were used for obtaining 3D images of elementary and technical hemp fibres.

3. Results and discussion

3.1. Tensile behavior

After Weibull analysis, the tensile strength of the different species ranges between 400 MPa and 920 MPa and the Young's modulus (E) from 30 GPa to 40 GPa. The comparison carried out by one-way ANOVA statistical analysis shows that among the different tested variants there is a significant difference in tensile strength between the 5 variants with lowest and the 5 with the highest mechanical properties, including strength and Young's modulus, at 95% confidence level.

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A typical curve obtained from a tensile test is shown in Figure 3. Three different types of stress-strain curves are observed after data analysis. Type I shows an initial stiffness increase followed by a stable zone where the Young's modulus remains until the break point (see Figure 3-a). Type 2 also exhibits an initial stiffness increase up until a maximum value is reached, then stiffness progressively reduces until the failure of the fibre (see Figure 3-b). Finally Type 3 curve shows an initial relatively high stiffness value, followed by a decrement of stiffness and a rapid increment until a stable value (see Figure 3-c).

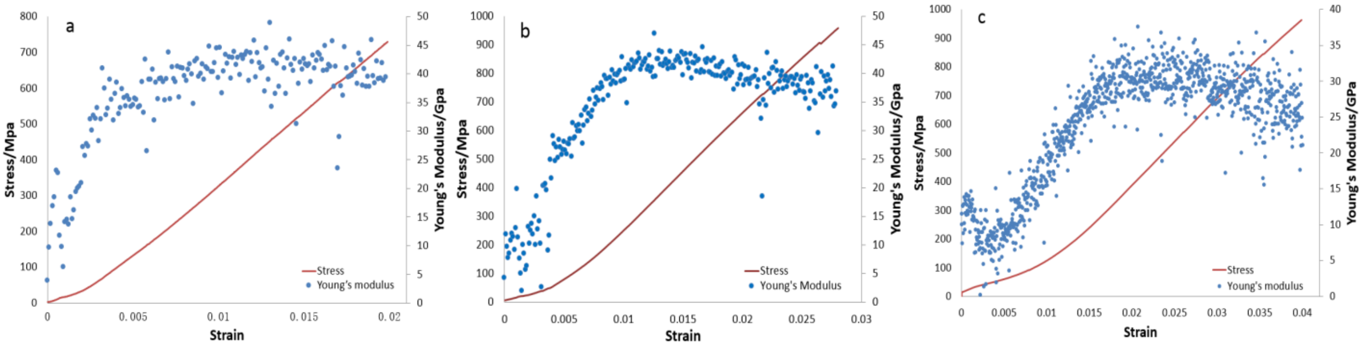

Figure 3. Typical stress-strain curves and variation of E modulus for the 3 different curve types.

Also according to ANOVA analysis, there is not a clear relation between the strength of the fibre and the type of curve. However, it could be observed that variants with the highest strength values and the variants with the lowest strength values show a significant proportion of type 3 and type 2 stress-strain curves respectively. Type 1 fibres are equally distributed among all variants.

3.2. Strain mapping

As it can be seen in Figures 4 and 5, the tensile behavior of hemp fibres is not uniform over the sample length, presenting a complex and very irregular pattern of strain concentrations. This might be caused by the interaction of elementary fibres which compose the technical fibre.

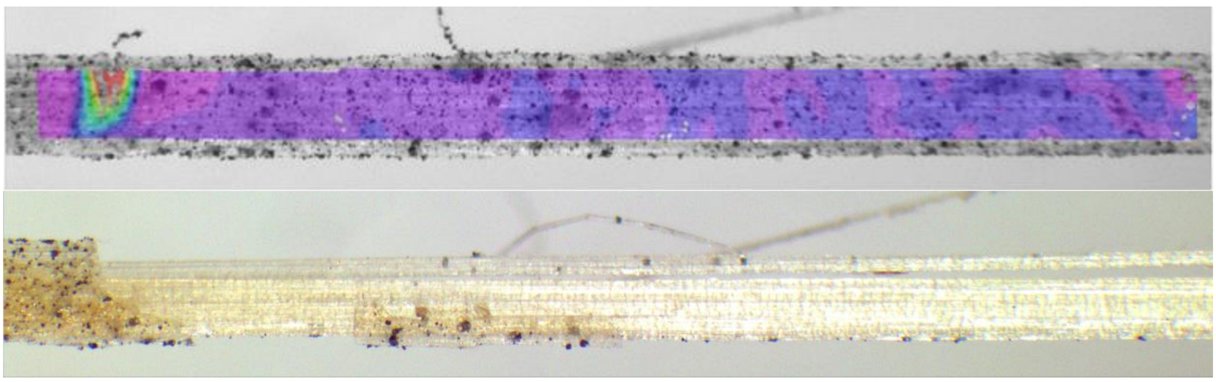

Figure 4. Strain mapping of a hemp fibre. Up: Strain pattern just before fracture, Down: Broken fibre.

Figure 4 shows a fibre with a high strain concentration on the left side, which is the point where the fibre finally breaks (see Figure 4-b). This validates the methodology which is able

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to detect minor deformations and cracks that eventually lead to the fibre breakage. However, it is not always the case that the final failure of the fibres occurs necessarily at the highest strain level location. The main disadvantage of the strain mapping technique is that it is only restricted to the outermost surface layer.

In order to correlate the fibre mechanical behavior with the strain evolution at a local microscale, strain mappings of weak and strong hemp fibres were analyzed. For each sample, 6 strain maps with increasing strain are displayed, see figure 5. The selected strain levels are 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, and the strain value just before fracture.

The strain maps show a homogeneous strain pattern at the beginning of the tensile test (see Figure 5) for both high and low strength fibres. However at medium and high strain levels, the strain pattern for high strength fibres becomes heterogeneous, showing areas of high and even apparent negative strain. These negative values are a consequence of the relaxation of some areas due to breakage of elementary fibres at the surface. On the other hand, strain patterns at medium and high strain levels remain relatively homogenous for low strength fibres. In general, strong hemp fibres show a more complex tensile behavior with several strain concentration spots.

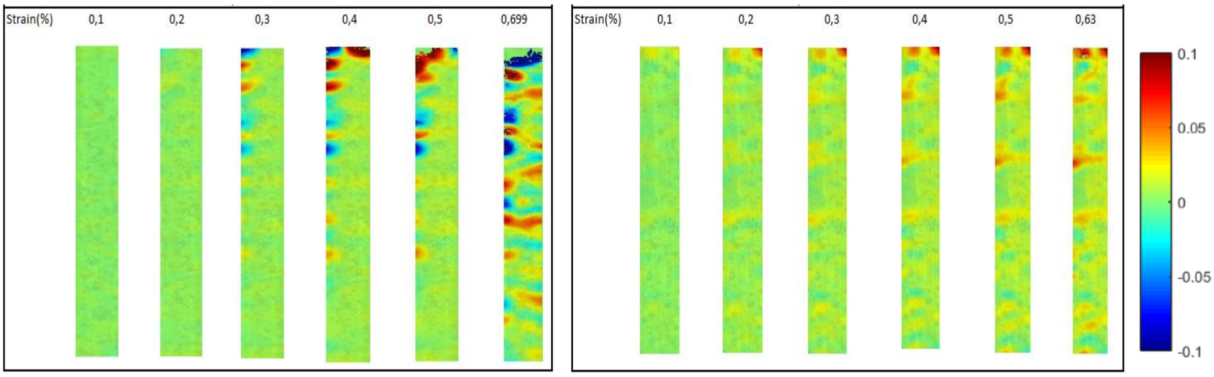

Figure 5. Strain maps of a typical high strength (left) and low strength (right) hemp fibre, showing the strain evolution during tensile loading.

3.3. Tomography

3D images of hemp fibres with high and low strength were analyzed in order to correlate the mechanical behaviour with the structure of the fibre. So far it has not been possible to make a direct connection, since this is ongoing work. However, it was possible to scan hemp fibres at different levels (see Figure 6) with a maximum resolution of 800 nm.

Tomography analysis allows to reveal solids with different density, as it is shown in Figure 6. This allows also to differentiate and characterize the different elements that compose a hemp fibre.

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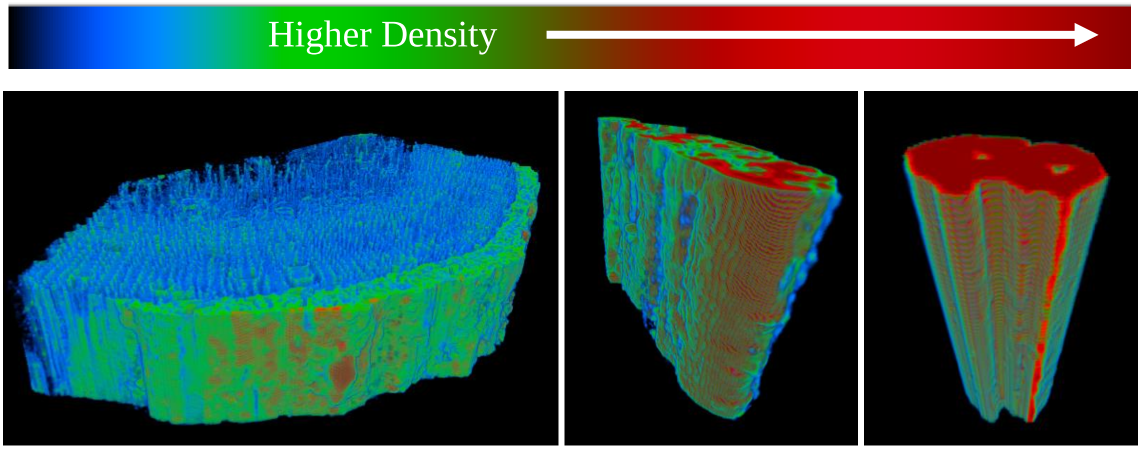

Figure 6. Three-dimensional reconstructions of a stem, technical, and elementary hemp fibres.

4. Conclusions

This study examined the tensile behavior of hemp technical fibres. In order to conduct both global and local strain analysis, standard tensile tests and digital image correlation were combined. The results revealed three different types of stress-strain curve. Accordingly, variants with the highest strength values and the variants with the lowest strength values show a significant proportion of type 3 and type 2 stress-strain curves respectively, and type 1 fibres are equally distributed among all variants.

The local strain patterns were found to be heterogeneous, but with remarkably more strain concentrations in the case of fibres with high strain values.

Acknowledgments

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References

[1] C.A. Fuentes, L.Q.N. Tran, C. Dupont-Gillain, W. Vanderlinden, S. De Feyter, A.W. Van Vuure, I. Verpoest, Wetting behaviour and surface properties of technical bamboo fibres, Colloids and Surfaces A: Physicochemical and Engineering Aspects 380(1-3) (2011) 89-99.
[2] T. Berfield, J. Patel, R. Shimmin, P. Braun, J. Lambros, N. Sottos, Micro-and nanoscale deformation measurement of surface and internal planes via digital image correlation, Experimental Mechanics 47(1) (2007) 51-62.
[3] M. Mehdikhani, M. Aravand, B. Sabuncuoglu, M.G. Callens, S.V. Lomov, L. Gorbatikh, Full-field strain measurements at the micro-scale in fiber-reinforced composites using digital image correlation, Composite Structures 140 (2016) 192-201.

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