Industrial Hemp Transformation for Composite Applications: Influence of Processing Parameters on the Fibre Properties

Vincent Placet, Camille François, Arnaud Day, Johnny Beaugrand and Pierre Ouagne

Abstract The main objective of this collaborative work is to characterize the influence of the processing stages of industrial hemp on the fibre properties. Transformation processes well suited for composite reinforcing textiles are considered. The work focuses on the different stages along the transformation chain of hemp, from the straw retting to the preform manufacturing. The main highlight is the predominant influence of retting on the tensile properties of individual fibres after their mechanical extraction from the stalks. Regarding the secondary processing, different technologies such as spinning, and use of natural binder systems are also proposed to produce yarns and woven fabrics. The effect on these secondary processing technologies and their parameters on the fibre properties are also characterised. The results show that the first steps of processing (retting and decortication) have the greatest impact on the tensile strength of hemp fibres.

Keywords Industrial hemp · Hemp fibres · Mechanical processing
Retting · Mechanical properties

V. Placet (✉) · C. François
FEMTO-ST, CNRS/UFC/ENSMM/UTBM, Université de Bourgogne Franche-Comté, Besançon, France
e-mail: vincent.placet@univ-fcomte.fr

A. Day
Fibres Recherche Développement, Troyes, France

J. Beaugrand
INRA, URCA UMR 614 FARE, Reims, France

J. Beaugrand
Research Unit BIA UR1268, INRA, Nantes, France

P. Ouagne
ENIT, EA 1905 LGP, Tarbes, France

R. Fangueiro and S. Rana (eds.), *Advances in Natural Fibre Composites*,
https://doi.org/10.1007/978-3-319-64641-1_2

Introduction

Today, in Europe, the well developed and marketed plant fibre continuous reinforcements (tapes, roving, fabrics and so on) for composites applications (PFCs) are mainly based on flax fibres. Alternatives to current commercial flax-based solutions are now considered and mobilize additional resources, like hemp. Considering their intrinsic properties, hemp fibres have a great opportunities for market capture in composite for secondary structural applications. So far, the main applications for hemp fibres are in the fields of pulp and paper (55%), insulation (26%), thermoplastic polymer composites (14%) and mulch (2.7%) [2]. The absence of hemp continuous reinforcement that would be available at an industrial scale could be connected to the lack of knowledge and engineering, in particular when compared to flax. This deficit for hemp is assigned to less intensive research activity, to a less important industrial production (in term of volume) and to technological barriers, such as fibre separation and the alignment of fibres throughout the transformation process. However, the knowledge on hemp and the readiness of the related transformation technologies have been progressing fast for these last few years thanks to the development of major research projects at national and European scales.

Currently, the processes used to separate the different vegetal fractions of hemp are mainly derivatives from the paper and technical textile industries. Technical fibres are generally extracted from the stalks using mechanical processes and lines made up of milling and cleaning systems. Among the different milling tools that are classically used, hammer mills are the most common [12]. They are efficient and they lead to clean technical fibres with a low content of shives. However, they are also very aggressive for the fibres themselves. They can lead to damages within the fibre wall, which is detrimental for the mechanical properties of individual fibres. Hernandez-Estrada et al. [6] showed recently that these first stages of processing (in particular decortication) have a great impact on the formation of dislocations in the fibres. Meanwhile, the preservation of the integrity of the fibres through the transformation stages and up to the composite scale is a real challenge and requires an optimization of both the primary and secondary processing. Scutching and hackling processes, which are commonly used for flax are less aggressive and also allows long and aligned technical fibres (line hemp) to be obtained [1]. This is, for sure, a processing route that has to be more used for hemp in view of their integration in composite materials. However, it is still required to adapt the harvesting and scutching procedures and machines to deal with the height and diameter of the stalks that are significantly greater than those of flax [1, 4]. In this work, we propose to characterize the influence of lab/pilot transformation processes that are currently developed for composite hemp-based reinforcements. Separation processes limiting the amount of damage caused to the fibres and secondary processes minimizing the twisting level at the scale of yarns while maximizing their tensile strength are targeted.

Over the last two decades, and since the renewal of interest in plant fibres, only few researchers have investigated the impact of processing on the mechanical properties of plant fibres. It still remains an open question, in particular when considering innovative transformation technologies. For flax, Van de Weyenberg et al. [13] showed that consecutive decortication stages of fibres (scutching, hackling…) change tremendously the mechanical properties of composites performance. They attributed this change to a modification of the mechanical and biochemical properties of the fibres. Thygesen et al. [15] studied the relationship between processing of cellulosic fibres (both hemp and flax) and fibre bundle strength. They studied processing methods that are traditionally applied for yarn production and also included retting, scutching, carding, and cottonization. They highlighted a monotonically decreasing relationship between the strength of fibre bundles and the number of processing steps, with an average reduction by 27% per processing step at the applied conditions. No large change in cellulose content and crystallinity were observed. So authors concluded that the reduction in strength must be explained by other changes in the fibres bundle ultrastructure. Indeed, in a bundle of fibre, the mechanical behaviour is controlled both by the performance of single fibres but also by the shear deformation at the interface between the fibres.

So, the overall objective of this work is to assess the influence of processing stages on the mechanical performance of single hemp fibres. The influence of processing parameters, including the retting level (an optional pre-processing parameter of the primary processing of stalks), extraction, combing, drawing and spinning on the mechanical properties of single hemp fibres are characterized. The gathering of results is the fruit of collaborative projects between several French labs.

Materials and Methods

Plant Material

Within this work, two types of materials were used to characterize the influence of processing on the mechanical properties of fibres, i.e. hemp stalks and combed fibres.

Hemp Stalks

The hemp stalks were supplied by La Chanvrière, a company in Bar sur Aube, France. Plants (Cannabis *sativa* L., cultivars ‘Fedora 17’) were cultivated in Laubressel in 2014 (GPS coordinates: 48° 18′ 00.1″N and 4° 14′ 12.0″E) on a chalky clay soil. Once mown, the straw was windrowed, turned over once, and laid in swatches on the ground for retting. Stalks were harvested after three different field retting times, 10, 39 and 75 days leading to three retting levels, noted R1, R2 and R3 respectively. For R3, stalks were turned over a second time, 60 days after moving, to homogenize retting. After reaching the targeted retting level, straws were balled and then stored under cover to prevent wetting. Hemp stalks used for experiments were collected at the same time, 136 days after mowing.

Hemp Sliver Laps

Long hemp fibres in the form of combed sliver laps were provided by an Italian company. Before being combed, the hemp fibres were carded and drawn. The slivers were then grouped together by a lap former to increase their size before being combed. The linear mass of the sliver laps was about 15,000 tex (14759 ± 613 tex). The length and diameter of single and technical fibres were measured on 30 samples randomly selected. The mean lengths of the single and technical fibres are 25 ± 14 and 83 ± 50 mm respectively. The mean diameters of the single and technical fibres are 20 ± 4 and 111 ± 33 μm.

Primary Processing of Hemp Stalks

Hemp technical fibres were extracted from the stalks by mechanical processing. This step was carried out by FRD® (www.f-r-d.fr) on a line made up of both milling and cleaning systems. This technology, developed at a pilot scale, is closed to flax industrial process. The same fibre extraction parameters were used for each sample. The setting of these parameters has been achieved through experiments done for several years and is considered as the best compromise between the fibre quality and the production yield. Fibre quality was evaluated with industrial standards developed by FRD, and by determining the residual content of shives in technical fibres and by measuring the fibre fineness.

The fibres extracted using this mechanical process are respectively named R1-MP, R2-MP and R3-MP depending on the initial retting level of the straws. The different stages and ways used to process the straws are schematically described on Fig. 1.

Secondary Processing of Hemp Fibres

An industrial drawing machine, adapted to long fibres, was used to reduce the linear mass of the sliver laps. Three drawing steps were used to obtain slivers with a linear mass of about 500 tex. After the first, second and third drawing steps, slivers with the following linear masses were respectively obtained: 3540 ± 139, 2204 ± 96 and 519 ± 49 tex. The slivers, which are assemblies of aligned fibres of finite length, do not possess generally enough cohesion to sustain further processing steps such as the textile architecturation (weaving, braiding…). So, they are generally

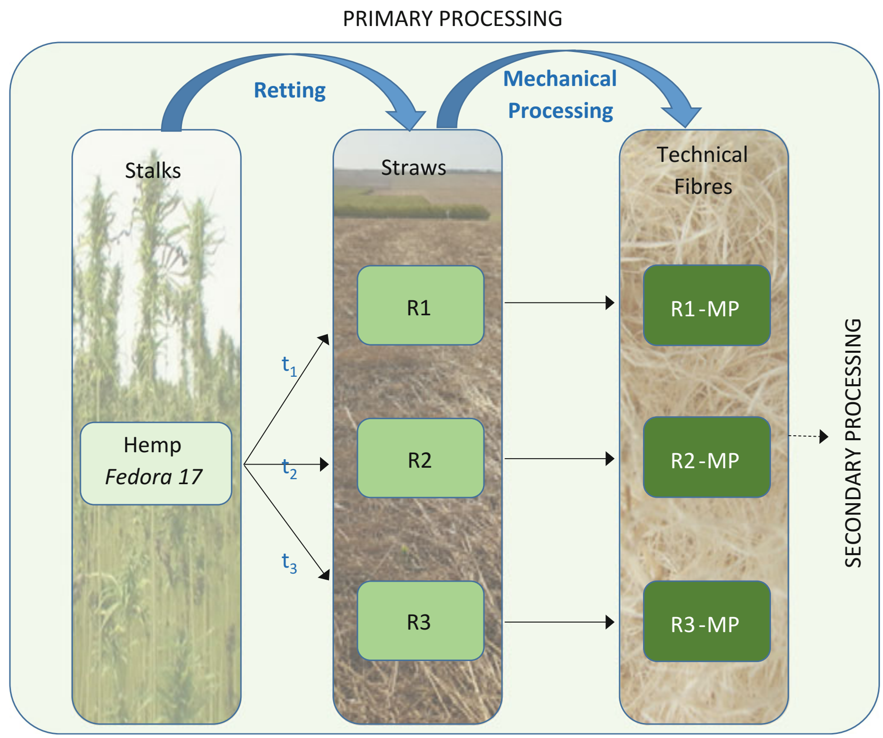


Fig. 1 Schematic representation of the main and different process stages used in this study for the fibre extraction

transformed into yarns. In this work, slivers were first transformed into non-twisted yarns (Y-NT, see Fig. 2). The fibres are slightly entangled during the drawing process. The failure load of the non-twisted yarns was 7.4 ± 1.9 N. This value is not high enough to manufacture a woven fabric or a braid. Thus, to increase the cohesion and the tensile performance of the non-twisted yarn, three other types of yarns were manufactured. Non-twisted yarns were bound with a natural adhesive constituent (Y-NTB). Low twisted unbound and bound with a natural adhesive constituent yarns were also manufactured (named respectively Y-LT and Y-LTB). Their load to failure were respectively: 19.2 ± 4.4, 19.7 ± 2.7 and 68.6 ± 18.6 N. A minimum of 15 N is generally required to manufacture woven fabrics, and the three types of yarns were therefore used for further processing steps.

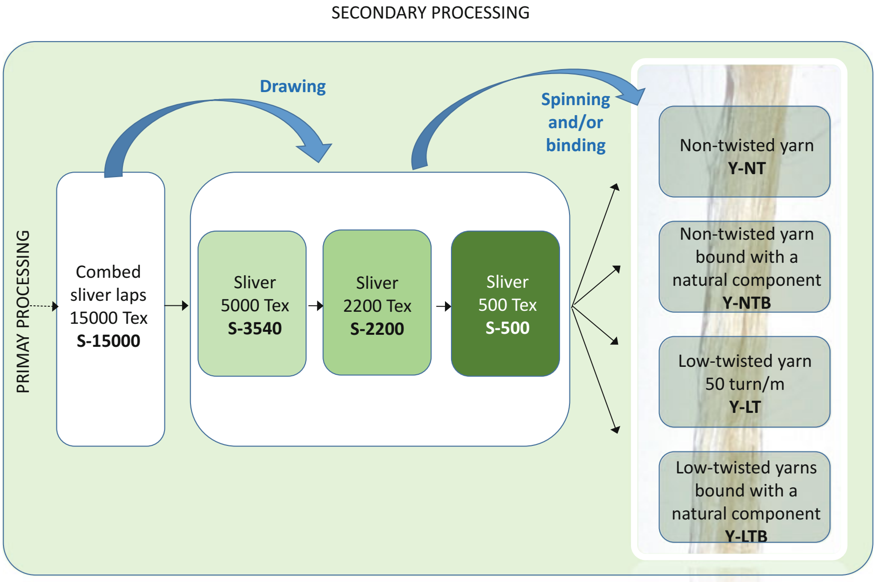


Fig. 2 Schematic representation of the secondary processing of hemp fibres, with the different drawing steps and spinning and/or binding solutions proposed in this study

Tensile Characterisation of Fibres

For each batch of fibres, thirty to fifty single fibres were manually extracted from the technical fibres using tweezers. They were glued onto thin paper support using adhesive (Loctite super glue) to facilitate their handling and examination prior to tensile testing. An electrodynamic machine (DMA Bose Electroforce 3230) was used to perform the monotonic tensile tests on fibres up to failure. The average width of each fibre was computed by obtaining ten measurements along its length using polarised light microscopy (Nikon Eclipse LV 150). The average width of each fibre batch was in the range of 20–25 μm. The paper frame supporting each fibre was clamped onto the testing machine and cut prior to the beginning of the tensile test. The clamping length was 10 mm. Fibres were tested at a constant crosshead displacement rate of 5 $\mu m\ s^{-1}$. The tensile tests were carried out at a temperature of 21 ± 2 °C and a relative humidity of 50% ± 5%. The applied force was measured using a 20 N load sensor, with a resolution of approximately 1 mN, and the displacement was measured using a LVDT with a resolution ranging from 0.1 μm. The sample elongation and the load were recorded continuously and the longitudinal mechanical properties (Young's modulus, ultimate strength and failure strain) of isolated hemp fibres determined. The strain was determined using the displacement measurements and the initial length of the fibre. The tensile stress was determined using the cross-section of each fibre and the apparent tangent modulus (E) was computed from the linear section of the first part of the stress–strain curve.

The effective cross-section area of the fibre was determined using the mean external diameter, assuming that the fibre is perfectly cylindrical and the lumen neglected. The mean values and standard deviation of these tensile properties were computed for each batch of fibres. Considering the highly skewed distribution that is generally observed for the tensile properties of plant fibres, a statistical analysis was also performed. The best distribution function for each property was identified.

Results and Discussion

Influence of Retting on the Tensile Properties of Fibres After Their Mechanical Extraction

Table 1 presents the values of bast fibre content and residual content of shives obtained after fibre extraction using the proposed mechanical process. The overall total bast fibre content in the straw is respectively 40.5, 50.7 and 42.5% for R1, R2 and R3. These high fibre contents contrast with data of literature that report a mean bast fibre content in hemp straw of approximately 30% (w/w) [7]. The field retting of straws could explain these high values. Indeed, ground retting lead to decohesion of stem tissues and decomposition of shives. The residue of shives can remain on the land after turning over and balling straw, inducing an increase of the relative content of bast fibres in straws.

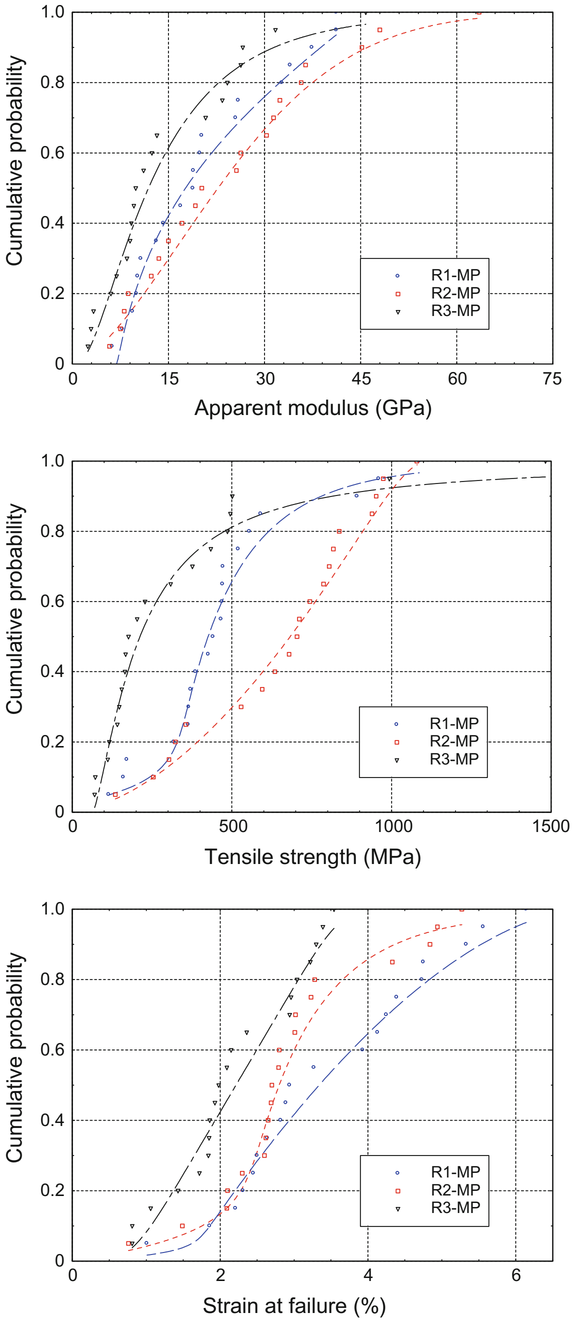
The percentage of residual shives in bast fibres after mechanical extraction decreases slightly (from 7.75 to 5.35%) with the increasing level of retting. This result underlines once more the influence of retting on decohesion of tissues within the straw.

The results from the tensile tests on single fibres after their extraction from the straws (at the three different retting times) are shown in Fig. 3. The same settings of the mechanical processing were used after the retting step for each of the three batches. No significant difference can be noticed on the apparent elastic modulus (Table 2). The mean value is comprised for each retting level between 15.5 and 17.5 GPa, with a similar and high standard-deviation of approximately 11 GPa. A significant decrease of the strain at failure can be observed with the increasing level of retting, from 3.5% for R1-MP to 2.2% for R3-MP. This result was already observed by Placet et al. [10] in a previous study. As far as the tensile strength is

Table 1 Bast fibre content and residual content of shives after fibre extraction for each batch of straws

	Bast fibre content (%)	Shive content (%)	Residual content of shives in bast fibres after extraction (%)
R1	40.5	59.5	7.75
R2	50.8	49.2	5.45
R3	42.5	57.5	5.35

Fig. 3 Tensile properties of single hemp fibres after mechanical extraction and for the three retting levels. *Markers* experimental data, *Dash lines* identified cumulative distribution functions



concerned, results show that the intermediate retting level (R2) leads to the highest value. Indeed, using these retting and primary processing settings (R2-MP), a tensile strength of 660 ± 265 MPa is measured at the scale of single fibres. This value is significantly higher to those measured when the stalks were lower or higher retted. Tensile strengths of 480 ± 245 and 340 ± 335 MPa were measured respectively for R1-MP and R3-MP. For R1-MP, the short time of exposure of the straws to the ground and thus to the microorganisms certainly does not permit the pectic content to be significantly attacked and removed by bacterial and fungal effectors. As a consequence, the bonding between the bast fibres both to each other and to the other constituents of the stalk remains strong. The further mechanical processing stage, aiming at separating the bast fibres from the ligneous shives, is aggressive and presumably highly damaging for the fibres. The damage formation can be directly related to the decrease in strength. Hernandez-Estrada et al. [6] showed recently that the first stage of processing (in particular decortication) has a greatest impact on the formation of damage. Hänninen et al. [5] also pointed out for flax that industrially processed flax fibres contain significantly more defects than green or retted ones.

Conversely, for R3-MP, it could be hypothesised that the fibre could have been processed from straws that had been over-retted. In addition to the removal of the pectic substances, the prolonged exposure time to the bacterial and fungal effectors could have presumably led to a degradation of the other constitutive polymers. This could induce a decrease in the mechanical performance of the fibres even before the mechanical processing. Placet et al. [10] showed that for over-retted straws, the decrease in the tensile performance of fibres can be attributed to a decrease in the crystalline cellulose index and to the degradation of hemicelluloses (a decrease in the relative content of xylose), which are thought to play an important role in the 3D organisation of wall macromolecules and the resulting mechanical properties. Liu et al. [8] also showed that a negative effect of field retting occurred after extended field retting (70 days in their retting conditions). This was attributed to the acceleration in the degradation of cellulose by the action of microorganisms.

The scattering in the tensile properties of R2-MP is high but expected and well-documented for plant fibres. The coefficients of variation (CoV) are on the same order of magnitude as generally measured for hemp fibres and other plant fibres such as jute, with a value of approximately 40–45% for the tensile rigidity and strength and 30–40% for the strain at failure [14]. This difference between the CoV in strain at failure on the one hand and rigidity and strength on the other hand is generally explained by the error when determining the effective cross-section of the fibres. Regarding R1-MP and R3-MP, the scattering in the strength values are very high with a respective CoV of 0.51 and 0.98. These values and the features of the distribution laws confirm that the mechanical processing on low-retted straws on the one hand, and over-retting on the other hand induce an increase in the number of flaws and flaw populations inside the fibres.

These results clearly indicate that field retting has to be perfectly tailored and controlled. If not, retting can be clearly detrimental for the quality of industrial hemp bast fibres and particularly for their mechanical properties. It is suggested that

Table 2 Tensile properties of single hemp fibres after mechanical extraction and for the three retting levels

	R1-MP	R2-MP	R3-MP
Apparent rigidity (GPa)	15.7 ± 11.6 0.9–41.5	17.5 ± 11.2 2.6–45.5	16.6 ± 12.7 2.9–43.3
Strain at failure (%)	3.5 ± 1.4 1.0–6.1	3.0 ± 1.1 0.8–5.3	2.2 ± 0.9 0.8–3.5
Tensile strength (MPa)	480 ± 245 115–1085	660 ± 265 135–1080	340 ± 335 70–1480
Diameter (μm)	25.4 ± 4.9 17.7–39.9	26.4 ± 5.3 17.0–36.0	28.9 ± 5.8 17.5–39.9

Mean ± SD, min…max values

an optimized period and level of field retting may be adopted to extract the fibres as gently and efficiently as possible.

Influence of Secondary Processing on the Tensile Properties of Fibres

Figure 4 and Table 3 synthesise the tensile properties measured on different batches of single fibres taken in yarns after their processing using the different technologies.

Fibres which were taken from the combed sliver lap (S-15000) have a mean rigidity of 18.2 ± 7.2 GPa, a strain at failure of 2.05 ± 0.8% and a strength of 325 ± 170 MPa. It can be observed that the three drawing steps applied to obtain slivers with a linear mass of about 500 tex induce a decrease of about 25% on the mean tensile rigidity and an increase in the average strain at failure of about 35%, without any significant change in strength. So, drawing appears to induce a softening of the longitudinal elastic properties of the single fibres. More surprisingly, twisting (at this level of approximately 50 turn/m) induces a slight increase in the tensile strength of single fibres, whether the yarn was treated with a natural adhesive or not, reaching an average value of 380 ± 180 and 395 ± 195 MPa for Y-LT and Y-LTB respectively. The origin of these phenomena (rigidity softening or hardening, increase or decrease on strength) observed under mechanical processing was not identified during this study and will be investigated in a forthcoming work by studying the microstructure of the fibres, in particular by measuring the cellulose crystallinity index and the cellulose microfibrils angle. Effectively, it is well known that the mechanical behaviour of wood and plant fibres are highly sensitive and dependent on their thermo-hygro-mechanical history [3, 9, 11]. So, it is not surprising that the transformation process influences their mechanical behaviour, since it involves various loading paths. One of the main difficulty, which researchers and industries are facing, is related both to the complexity of the transformation stages and the complexity of the thermo-hygro-mechanical behaviour of plant fibres. They

Fig. 4 Tensile properties of single hemp fibres after secondary processing. *Markers* experimental data, *lines* identified cumulative distribution functions

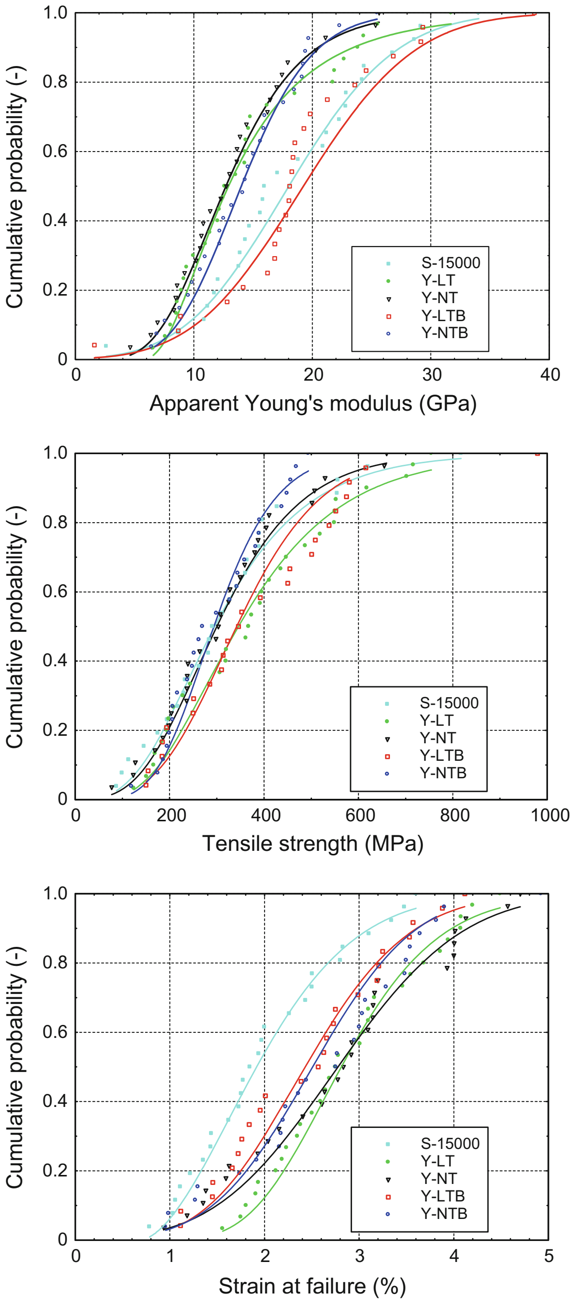


Table 3 Tensile properties of single hemp fibres after secondary processing

	S-15000	Y-NT	Y-NTB	Y-LT	Y-LTB
Apparent rigidity (GPa)	18.2 ± 7.2 2.6–34	13.5 ± 5.4 4.6–25.6	14.4 ± 4.8 6.4–25.5	14.7 ± 6.3 6.6–31.7	18.9 ± 7.6 1.6–38.7
Strain at failure (%)	2.05 ± 0.8 0.8–3.6	2.8 ± 1 0.9–4.7	2.7 ± 1 0.9–4.9	2.9 ± 0.8 1.6–4.5	2.5 ± 0.9 1.1–4.1
Tensile strength (MPa)	325 ± 170 90–820	320 ± 150 75–660	305 ± 105 120–495	380 ± 180 125–755	395 ± 195 150–980
Diameter (μm)	20.2 ± 4.5 13.2–35.2	21.5 ± 5 13.2–33	22.2 ± 4.5 14.6–33.2	24.7 ± 5 17.3–37.4	22.3 ± 4.4 14.8–32.9

Mean ± SD, min…max values

exhibit in particular couplings and stiffening phenomena under mechanical loading and/or environmental exposure that are still not fully understood [11]. It is also highly difficult to predict the loading paths and levels endured by the fibres during the processing stages, even if well mastering the process settings.

It can also be noticed that the treatment with a natural adhesive lead to an increase in the tensile rigidity. A mean value of 18.9 ± 7.6 GPa was measured for Y-LTB. An interaction between the natural adhesive and the constitutive polymers of the fibre wall can be assumed. Biochemical and surface analyses have to be done to confirm this hypothesis.

In general, it can be concluded that, even if secondary process stages can influence the tensile performance of single fibres, the chosen technologies have a lower impact than extraction step.

Conclusion

This work focused on the impact of the hemp transformation stages, from retting to yarn manufacturing, on the tensile behaviour of single fibres. Results emphasize the predominant role of retting on the further aggressive mechanical fibre separation steps and on the quality of the resulting fibres. It also points out the necessity to tailor the field retting to ensure a good quality and properties stability at the scale of industrially processed fibres. In light of these results, it can already be concluded that it should be essential to dispose of a tool indicating how to manage straw quality and retting before the mechanical processing of high-grade fibres. Further processing steps towards hemp reinforcement (such as drawing, spinning, binding with natural adhesive) can also influence the strength and rigidity of fibres but with a lower impact.

Acknowledgements Johnny Beaugrand is thankful for the support provided by the MATRICE state-to-country (Région Alsace Champagne-Ardenne Lorraine) program, France. Arnaud Day would like to thank, ADEME, Troyes Champagne Métropole and Région Alsace Champagne-Ardenne Lorraine for their financial support. The authors would also like to thank Groupe Depestèle (France) for their technical support and the use of their drawing and binding facilities.

References

1. Amaducci, S., & Gusovius, H. J. (2010). Hemp-cultivation, extraction and processing. In J. Müssig (Ed.), *Industrial applications of natural fibres—Structure, properties and technical applications.* Wiley series.
2. Carus, M. (2015). World-wide market data on hemp and other bast fibres—Status and outlook. In *12th International Conference of the European Industrial Hemp Association (EIHA), Wesseling, Germany*, May 20–21, 2015.
3. Guicheret-Retel, V., Cisse, O., Placet, V., Beaugrand, J., Pernes, M., & Boubakar, M. L. (2015). Creep behaviour of single hemp fibres. Part II: Influence of loading level, moisture content and moisture variation. *Journal Materials Science, 50*(5), 2061–2072.
4. Gusovius, H. J., Hoffman, T., Budde, J., & Lühr, C. (2016). Still special? Harvesting procedures for industrial hemp. *Landtechnik, 71*(1), 14–24.
5. Hänninen, T., Thygesen, A., Mehmod, S., Madsen, B., & Hughes, M. (2012). Mechanical processing of bast fibres: The occurrence of damage and its effect on fibre structure. *Industrial Crops and Products, 39,* 7–11.
6. Hernandez-Estrada, A., Gusovius, H.-J., Müssig, J., & Hughes, M. (2016). Assessing the susceptibility of hemp fibre to the formation of dislocations during processing. *Industrial Crops and Products, 85,* 382–388.
7. Legros, S., Picault, S., & Cerruti, N. (2013). Factors affecting the yield of industrial hemp—Experimental results from france. In P. Bouloc, S. Allegret, & L. Arnaud (Eds.), *Hemp: Industrial production and uses*. doi:10.1079/9781845937935.0000.
8. Liu, M., Fernando, D., Daniel, G., Madsen, B., Meyer, A. S., Ale, M. T., et al. (2015). Effect of harvest time and field retting duration on the chemical composition, morphology and mechanical properties of hemp fibers. *Industrial Crops and Products, 6,* 29–39.
9. Madsen, B., & Gamstedt, K. (2013). Wood versus plant fibers: Similarities and differences in composite applications. *Advances in Materials Science and Engineering*, ID564346, 14.
10. Placet, V., Day, A., & Beaugrand, J. (2017). The influence of unintended field retting on the physicochemical and mechanical properties of industrial hemp bast fibres. *Journal of Materials Science, 52*(10), 5759–5777.
11. Placet, V., Cisse, O., & Boubakar, L. (2014). Nonlinear tensile behaviour of elementary hemp fibres. Part I: Investigation of the possible origins using repeated progressive loading with in situ microscopic observations. *Composites Part A: Applied Science and Manufacturing, 56,* 319–327.
12. Sponner, J., Toth, L., Cziger, S., & Franck, R. R. (2005). Hemp. In R. R. Franck (Ed.), *Bast and other plant fibres*. Boca Raton, USA.
13. Van de Weyenberg, I., Ivens, J., De Coster, A., Kino, B., Baetens, E., & Verpoest, I. (2003). Influence of processing and chemical treatment of flax fibres on their composites. *Composites Science and Technology, 63*(9), 1241–1246.
14. Virk, A. (2010). *Numerical models for natural fibre composites with stochastic properties*. Ph.D. thesis, Plymouth University.
15. Thygesen, A., Madsen, B., Bjerre, A. B., & Lilholt, H. (2011). Cellulosic fibers: Effect of processing on fiber bundle strength. *Journal of Natural Fibers, 8*(3), 161–175.

http://www.springer.com/978-3-319-64640-4

Advances in Natural Fibre Composites
Raw Materials, Processing and Analysis
Fangueiro, R.; Rana, S. (Eds.)
2018, XII, 280 p. 146 illus., 98 illus. in color., Hardcover
ISBN: 978-3-319-64640-4