FISEVIER

Contents lists available at ScienceDirect

Composites Part A



journal homepage: www.elsevier.com/locate/compositesa

Analysis of the potential of hemp fibres for load bearing composite reinforcement using classical field management techniques and carded route

Marie Grégoire ^a, Mahadev Bar ^a, Xavier Gabrion ^b, Gilles Koolen ^c, Salvatore Musio ^d, Debora Botturi ^e, Giorgio Rondi ^e, Stefano Amaducci ^d, Emmanuel De Luycker ^{a,*}, Aart Van Vuure ^c, Vincent Placet ^b, Pierre Ouagne ^a

^a Laboratoire Génie de Production, LGP, Université de Toulouse, UTTOP, 47 avenue d'Azereix, F-65016 Tarbes, France

^b SupMicroTech, Université de Franche-Comté, CNRS, Institut FEMTO-ST, Besançon, France

^c Department of Materials Engineering, KU Leuven, B-3001, Heverlee, Belgium

^d Department of Sustainable Crop Production, Università Cattolica del Sacro Cuore, Piacenza, Italy

^e Linificio e Canapificio Nazionale, Villa d'Almè, Italy

ARTICLE INFO

Keywords: Natural fibres Hemp fibre Fibre properties Mechanical testing Fibre processing

ABSTRACT

An alternative route to the traditional scutching and hackling processes was tested to produce hemp fibres suitable for load bearing composites. A classical approach consisting of a succession of breaking roller and breaking card, was used. The morphology and mechanical properties of the fibres were characterised. The tensile properties after breaking card, extra finishing card and combing were comparable to those obtained from the traditional approach. This similarity may be attributed to the drawing process which serves to homogenise the fibre properties by mitigating the number and severity of structural defects. This, combined with the possibility of using a more flexible approach than scutching and hackling may present an opportunity to increase the European production of technical fibres for load bearing applications. This would satisfy industries seeking large quantities of high potential fibres, a demand that cannot be adequately met by the textile flax resources which are increasingly diverted to the garment industry.

1. Introduction

For environmental reasons, composite materials incorporating biosourced components, in particular plant fibres, have been developed and industrialized for some years [1]. Concept of life cycle engineering is used to quantify the performance of a product as well as its environmental and financial impacts [2]. Different types of plants can be used to manufacture 1D reinforcement products [3]. However, to date, the most widely used plant fibres for those applications are from textile flax. However, despite an increasing demand, expanding the production of this material for technical applications beyond niche markets is currently challenging, primarily due to the saturation caused by demands from the garment textile industry. The traditional textile flax growing areas are limited to North-West France, Belgium and the Netherlands, and their capacity for expansion in its current form is constrained. Hemp represents a promising complement to textile flax for load-bearing applications. It is a relatively easy crop to cultivate, requiring minimal inputs, and its by-products can be used in a number of areas [4,5]. Additionally, the plant produces dry stems yields of between 6 and 15 tonnes per hectare [6,7], which may be higher than the 5 to 7 tonnes per hectare of dry stems generally obtained for textile flax [8]. Consequently, comparable quantities of long hackled fibre can be obtained for flax and hemp (1.5 and 1.76 tonnes per hectare respectively) [7]. However, the stem and fibre yields observed depend on the varieties

* Corresponding author.

https://doi.org/10.1016/j.compositesa.2024.108658

Received 19 July 2024; Received in revised form 9 December 2024; Accepted 10 December 2024 Available online 15 December 2024

E-mail addresses: marie.gregoire@uttop.fr (M. Grégoire), mahadev.bar@uttop.fr (M. Bar), xavier.gabrion@ens2m.fr (X. Gabrion), gilles.koolen@kuleuven.be (G. Koolen), Salvatore.Musio@unicatt.it (S. Musio), d.botturi@linificio.it (D. Botturi), g.Rondi@linificio.it (G. Rondi), stefano.amaducci@unicatt.it (S. Amaducci), emmanuel.de-luycker@uttop.fr (E. De Luycker), aartwillem.vanvuure@kuleuven.be (A. Van Vuure), vincent.placet@univ-fcomte.fr (V. Placet), pierre.ouagne@uttop.fr (P. Ouagne).

¹³⁵⁹⁻⁸³⁵X/© 2024 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

under study, the agronomic parameters employed and the cultivation techniques applied [4]. The fibres extracted from hemp stems exhibit high tensile properties, rendering them suitable for utilisation in composite materials [7,9]. They can be extracted from hemp stems throughout a number of European regions, extending from the west to the east and thriving in wide range of soil types and climatic conditions [10].

Two principal categories of apparatus are employed for the extraction of plant fibres from hemp or flax stems. The first is the so-called "allfibre" route, which is traditionally used to extract hemp fibres using devices such as hammer mills, roller mills or ball mills [11]. The principal advantages of this extraction technique are its productivity and the fact that it does not require an ordered material at the input of the system. Hemp is frequently harvested with combine harvesters that do not favour an alignment of the stems at the output of the machine. However, these extraction techniques such as the traditional hammer mills are very aggressive and significantly reduce the mechanical and morphological properties of the extracted fibres [11,12]. Such fibres can then only be used as reinforcements in short-fibres injection-moulded composites.

In order to overcome the problems encountered when processing stems on an all-fibre machine, studies have recently been carried out on a scutching/hackling type long-fibre extraction device [7]. This traditional extraction route for textile flax has been successfully adapted when the extraction parameters are modified to suit hemp, with notable reductions in processing speeds compared to what is used for textile flax [13]. The long fibres obtained possess the requisite mechanical and morphological properties for use in load-bearing composite materials [9].

However, the long scutched and hackled hemp fibres, similar to those of textile flax, are in high demand from the garment textile industry. Consequently, the prices may be too high for large-scale technical applications. By-product fibres (flax tows) derived from the scutching and hackling processes have been successfully employed in the development of yarns with high mechanical properties, which can be incorporated into composite materials [14]. Once again, this kind of source is limited to regions where scutching mills are present with all the harvesting chain dedicated to the process. This type of system requires the input material (hemp stems) to be aligned and cut because hemp stems are higher than flax stems. The harvesting of the stems by combine machines, as is usually done for hemp, is therefore unsuitable. It requires the utilisation of specific hemp harvesting machines. Given that hemp can be cultivated across most of Europe, the use of scutching mills, which are concentrated in the north of France and Belgium, is not profitable for the hemp growers who are not situated in these areas. Consequently, they are forced to transport the stems to distant locations, which is both costly and environmentally unsustainable. This study therefore investigates the possibility of using an alternative extraction device that can accept at its input disordered straws harvested using traditional harvesting tools such as those used for hay, with only minor modifications. Such an extraction facility (smaller in size than a traditional scutching mill) could be placed near hemp growing areas, thus leading to a reduction in production costs for industrial hemp growers while allowing the production of fibres suitable for use in load-bearing composite materials.

In light of the aforementioned considerations, an investigation was conducted into the potential of an all-fibre extraction route comprising the use of breaking rollers and a breaking card. The impact of the extraction process on the mechanical and morphological properties of the long fibres obtained was studied to ensure compatibility with the requirements for use in load-bearing composite materials. The outcomes were then compared with those previously obtained through the industrial extraction of hemp fibres using a scutching/hackling device. Furthermore, a discussion was conducted on the impact of the extraction methods on the mechanical properties of the subsequently manufactured composites.

2. Materials and methods

2.1. Plant material

The hemp stems used in this study were grown within the framework of the European SSUCHY project by the Universita Cattolica del Sacro Cuore (UCSC, Piacenza, Italy). The hemp variety used, FUTURA 75, is a late monoecious variety developed in France by the company Hemp-it and intended for double cropping. It was thus possible to harvest both fibres and seeds simultaneously. The hemp was sown in April 2019 at a seed density of 70 kg per hectare before being harvested in early August, at the end of flowering. The resulting stems were retted in the field for a period of three weeks, after which they were cut into one-metre-long sections. The extractions were then carried out on the sections from the lower part of the stems.

2.2. The fibre extraction routes

Two extraction routes were used to process the hemp stems and thus compare the obtained fibre quality properties (Fig. 1). The material batches corresponding to each route at different stages are indicated in Table 1.

2.2.1. Scutching and hackling extraction

An industrial scutching/hackling device located in the premises of the company "Terre de Lin" in Normandy (Saint-Pierre-Le-Viger, France) (Fig. 2) was used. Initially considered for processing textile flax, it can also be used to extract fibres from aligned hemp stems. It is composed of two distinct modules:

– The first module, developed by Depoortere (Waregem, Belgium), comprises a breaking and scutching apparatus (Fig. 3). The device is used to break up the wood part of the stems and to extract the shives and vegetal dusts obtained, which would have remained trapped in the



Fig. 1. Schematic representation of the different extraction methods and steps used in this study and of the batches of fibres sampled at the output of the different methods.

Table 1

Name of the material batches considered and associated process.

Batch Name	Route	Finishing Card/ Combing	Drawing
Sc	Scutching/Hackling		
ScD	Scutching/Hackling		X
Ca	Breaking Rollers/Breaking		
	Card		
CaD	Breaking Rollers/Breaking		X
	Card		
CaCoD	Breaking Rollers/Breaking	X	X
	Card		



Fig. 2. Industrial scutching/hackling extraction device available at Terre de Lin.

fibres. The breaking process is conducted via a series of horizontal, counter-rotating pairs of fluted rollers with progressive increase in the number of teeth. The scutching is then carried out in the drum system composed of a succession of pairs of rotating blades, each of them rotating in an opposite direction. When used for processing flax stems, this device is capable of processing up to one tonne of the aforementioned material per hour, with a yield of up to 250 kg of scutched long fibres.

- The second module is an independent Linimpianti hackling machine (Linificio, Villa d'Almè, Italy) for progressive hackling. This device can process up to 80 kg of scutched fibre per hour.

The extraction parameters used were identical to those used in the industrial extraction of textile flax fibres (about 200 turns/min for the turbines and cadencies of about 2000 kg/h of straw processed).

The fibres were assembled into slivers at the output of the hackling system and a first batch of fibres was taken out (Sc). The rest of the material was then drawn to form a sliver and roving type yarns using a long fibre device developed by the company Linificio (Villa d'Almè, Italy) (ScD). The operating principle of this device will be explained in the section 2.3.

2.2.2. "All-fibre" extraction

The impact of an "all-fibre" extraction route on the mechanical and morphological properties of the fibres was studied in a second time. The fibres were extracted using a device developed by the company Hemp-Act (Hemp-Act, France) and available at their premises. The apparatus comprised a succession of pairs of grooved rollers, which permitted the simultaneous breaking and shaking of the hemp stems which were processed twice through this first module. After breaking, the stems were passed three times through a breaking card. A sliver of fibres was obtained at the end of the carding process and constitutes Ca. A more comprehensive description of this device can be found in the work of [15]. An additional sample of fibres was obtained following a drawing stage identical to the one applied previously to the scutched/hackled fibres (CaD).

The sliver of fibres obtained after the three passes through the breaking card exhibited a high degree of regularity, but visual observation revealed the presence of a small quantity of shives trapped within the fibres. To eliminate these pieces of wood which could potentially present a challenge for the subsequent manufacture of composite materials, an alternative extraction route was investigated. After a first pass through the breaking card device presented earlier, the hemp fibres were then processed in a finishing carding machine before being combed in a tow-combing tool specially adapted to bast fibres from the company NSC (n. Schlumberger) and included in the chain as illustrated in Figs. 1 and 4. The fibres were then drawn before a final batch of fibres (CaCoD) was taken.

2.2.3. The lab scale drawing and roving devices

The laboratory long fibre drawing device used in this study was developed by the Linimpianti company (Linificio, Villa d'Almè, Italy). It transforms a sliver of about 15,000 tex to a lower size sliver of 150 tex in 6 drafting/doubling stages using gill-type systems arranged in parallel. Each gill system has an increasing tooth density over the 6 passes.

This process has for objective to reduce the size of the sliver and to homogenise the fibre length and diameters during the 6 drawing/ doubling steps. At the end of the drawing process, the fibres were transformed into roving type yarns using a specific bench adapted to bast fibres. During this process, a low twist was applied to the fibres (\sim 30 turns/m).

2.3. Morphological analysis of the fibre bundles

2.3.1. Determination of the diameters of the fibres bundles

A morphological analysis of the fibre bundles is crucial for determining the impact of the different extraction steps on their level of



Fig. 3. Schematic representation of the extraction stages by industrial scutching/hackling (from left to right).



Fig. 4. Schematic representation of the extraction stage by carding route (from right to left).

separation. As indicated in the literature, this parameter has a significant impact on the quality of the composites manufactured subsequently [16,17].

In this study, the FiberShape device developed by IST AG (Vilters, Switzerland) was used to measure the width of the fibre bundles. Assuming a circular cross-section, this width corresponds to the diameter of the fibre. This device consists of a high-precision scanner Reflecta MF 5000 (Reflecta, Eutingen im Gaü, Germany), combined with the fibre recognition software Silverfast developed by Lasersoft Imaging (LaserSoft Imaging, Kiel, Germany). The fibre bundles were trimmed into 2 cm long sections and positioned between two glass plates which were subsequently inserted into the high-precision scanner. The image was captured with an accuracy of 3200 dpi. The device is capable of measuring a large quantity of fibres in a relatively short time, with the capacity to process more than 10,000 fibres in a single measurement. Additionally, the device is capable of measuring fibres with large diameters (up to 1000 μ m).

The results are presented in the form of boxplots. The outer lines of the box represent the first quartile (25 %) denoted Q1 and the third quartile (75 %) denoted Q3; the line inside the box is the median (50 %). The ends of the segments from the boxes indicate the minimum and maximum values obtained if no outliers are present. Outliers are points outside the interval [Q1-1.5IQR, Q3 + 1.5IQR], with IQR the interquartile range equals (Q3-Q1). If outliers are present, they are represented with dots, the end of the segment on the same side of the box represent then (Q1-1.5IQR) and no longer the minimum value or Q3 + 1.5IQR and no longer the maximum value. Additionally, the mean value is displayed with a diamond. All the statistical data are weighted by the length of each measured object.

2.3.2. Determination of the lengths of the fibres bundles

The length of fibre bundles was determined using the FibreScanner device developed by IST AG (Vilters, Switzerland). The device can be utilised for the analysis of length distributions on any type of natural fibre. In order to achieve this, a set of fibre bundles was positioned in a succession of combs. A clamp grabbed the end of the fibres protruding from the combs and pulled them across a laser beam. The presence of fibres, highlighted by the laser in the focal plane of an optical sensor is detected up to the end of each fibre, enabling the accurate length measurement of the pulled fibres. Fibres of lengths between 2 cm and 40 cm can be measured. A minimum of 100 fibres were measured for each batch. Results are displayed with boxplot as described in the previous section. Data are also weighted by the length of each measured fiber.

2.4. Determination of the mechanical properties of fibres

In order to ascertain the influence of the extraction and refining stages on the mechanical properties of the fibres, 50 elementary fibres were randomly selected from each of the 5 batches. Subsequently, the fibres were subjected to tensile testing using a device developed by the company DiaStron (Dia-Stron Ltd., Hampshire, UK). The apparatus comprises two distinct modules, the operation of which is described in detail below. The tests were conducted under controlled conditions, with a temperature of 20 $^{\circ}$ C and a humidity level between 60 % and 65 % in the laboratory. Same definition is used for representing the datas with boxplots (with no weighting by the length).

2.4.1. Determination of the cross-section of the fibres

The individual fibres were manually separated without any preliminary treatment and then glued at both ends into plastic tabs using a light-curing adhesive (DYMAX, Wiesbaden, Germany). A gauge length of 12 mm was employed.

The Fibre Dimensional Analysis System (FDAS) by Diastron Ltd. (Hampshire, UK) and UV Win software were employed to scan the fibres mounted on the plastic tabs with the objective of determining their cross-section area using shadow projection measurement. The fibres were positioned and held in the rotating jaws of the FDAS system by means of a pneumatic mechanism. The width of the fibre (or more precisely, its projected shadow) was then measured at a minimum of 10 locations along the 12 mm gauge length using a high precision laser (LSM 500S, Mitutoyo, Japan). By rotating the fibre during the measurement, the maximum and minimum widths were recorded for each position. An elliptical model was then employed to calculate the fibre cross-section area. Indeed, the literature on fibre bundles indicates that this type of model produces results closer to the real cross-section of the fibre than a circular model [18,19]. The device enables the measurement of widths with an accuracy of 0.01 μ m.

2.4.2. Tensile testing of the fibres

Following the cross-section measurement, the single elementary fibres were subjected to tensile testing on the second modulus of the device in order to determine their mechanical properties, namely strength at break, Young's modulus and strain at break. The tensile modulus is calculated by taking two different deformation points in the linear zone at the end of the stress–strain curve.

A high-precision extensioneter (Lex 820, DiaStron Ltd., Hampshire, UK), with a load cell capacity of \pm 20 N, was employed for this purpose. This apparatus is suitable for the testing of fibres breaking at low strain. The displacement is achieved using a stepper motor which controls the displacement with an accuracy of 1 μ m.

The tests were conducted at a strain rate of 1 mm/min and a compliance correction was employed to account for the device rigidity.

2.5. Impregnated fibre bundle tests (IFBT)

2.5.1. Manufacture of the IFBT specimens

In order to ascertain the mechanical performance of the fibres within the composite material, Impregnated Fibre Bundle Tests were conducted.

For this purpose, six UD composite coupons were manufactured for each batch identified (i.e. 1 to 6). The methodology employed is based on ISO 10618: 2004 [20], taking into account the modifications for plant fibres as described in the technical document published by the European Confederation of Flax and Hemp (CELC) [21]. The hemp fibres were initially subjected to a drying process for a minimum of 24 h at a temperature of 60 °C, after which they were cut into approximately 200 mm length samples. The fibres were manually stretched so that they are as much as possible positioned parallel to each other's. They were then positioned in a mould of dimensions 200 mm x 10 mm with a spacer of 2 mm before being impregnated with GreenPoxy 56 resin from Sicomin, France. The quantity of resin utilised was quantified in order to achieve a fibre volume fraction of approximately 35 %. This fibre volume fraction was chosen with the objective to reach a good impregnation, a low porosity, a good spatial distribution of the fibres and a good control of the composite thickness as recommended in [20,21]. Subsequently, the IFBT specimens were subjected to a two-hour curing process at 150 °C and a pressure of 2 bars. A manual hydraulic press was employed and the pressure was asserted on the spacers.

Subsequent to fabrication, the specimens were maintained in a climatic chamber at a temperature of 23 °C and a relative humidity of 50 % for a minimum period of four weeks. The mechanical properties of the fibres were then back-calculated using a rule of mixtures (See Eq. [1;2]).

2.5.2. Tensile tests of the IFBT specimens

The six specimens were subjected to tensile testing on a Zwick/Roell Z100 universal testing machine equipped with a 100 kN force cell with a gauge length of 160 mm. The longitudinal strain was quantified utilising a clip-on extensometer with a gauge length of 50 mm. The tests were conducted with a speed rate of 2 mm/min.

The effective mechanical properties of the tested hemp fibres were determined by the inverse method between 0 % and 0.1 % of deformation, in accordance with the work of Bensadoun et al. [22]. The following equations (Eq. [1] and Eq. [2]) were thus employed to determine the mechanical properties of the fibres:

$$E_f = \frac{E_c - E_m (1 - V_f)}{V_f}$$
(1)

$$\sigma_f = \frac{\sigma_c - \sigma_m (1 - V_f)}{V_f} \tag{2}$$

With E_f the modulus of the fibres (GPa)

- E_m the modulus of the matrix (GPa).
- E_c the modulus of the composite (GPa).
- V_f the volumetric fraction of fibres.
- σ_f the effective longitudinal tensile strength of the fibres (MPa).
- σ_c the stress at failure of the composite (MPa).
- σ_m the stress in the matrix at the composite's failure (MPa).

2.6. Statistical analysis

Variance analysis (ANOVA) is performed in R software (aov function) to compare the different population weighted by the length or not. Tuckey test is then used (TukeyHSD function) to interpret the results with 95 % confidence.

3. Results and discussion

In order to meet the aim of this study, the carded fibre samples obtained are compared with what is already produced industrially by scutching/hackling and which is suitable for the production of roving type yarns [7].

3.1. Comparison of fibre bundle diameters

The results of the fibre bundle diameter measurements on the highprecision scanner are shown in Fig. 5. First of all, the median diameters weighted by the length obtained vary between 52.6 μ m (ScD) and 68.4 μ m (Ca). The analyses show that drawing leads to a significant reduction in fibre diameters (ScD VS Sc and CaD VS Ca) and therefore contributes to a significant refinement of the material. The drawn fibres of CaCoD obtained after passing through a finishing card and tow-combing have a significantly smaller diameter than the initial carded material (Ca) but no significant different diameters than the fibres from CaD.

The fibres are generally too coarse for use in the field of garment textiles, where fibre diameters of about 30 μ m or lower are required, but fine enough for use in composite materials. It is remarkable that both the carded and scutching/hackling extraction routes are equally performant



Fig. 5. Diameters of the fibre bundles taken at the different extraction stages (Batches), data are displayed weighted by the length; letters indicate the groups with no statistical differences.

in terms of fibre divisibility. However, the roving type yarns manufactured from the fibres of CaD contain small quantities of impurities such as pieces of bark and small pieces of shives, the wood part of the stems. This type of waste, trapped in the fibres, will be found in the composite materials manufactured afterwards and may have an impact on their mechanical properties Therefore, an additional cleaning step is performed (CaCoD). In this case, the fibres are free of shives and are therefore expected to be more suitable for the manufacture of composite materials. However, they show no significant decrease in diameter compared to the CaD fibres but show higher diameters than for ScD. This is probably due to the elimination of low diameter fibres during combing which fall off the machine as combing tows.

A study of the diameter diagrams of the fibre bundles of the different batches (Fig. 5) showed a type of distribution that is identical for the 3 batches after drawing as classically observed in the textile industry for flax [7]. Thus, the most represented fibres are those of small diameters, those of intermediate diameters being progressively and inversely present in smaller and smaller quantities. Fibres with large diameters (more than 200 μ m) are only present in very small quantities.

Looking in more detail at the diameter distributions of the bundles (Fig. 6), it appears that the additional carding and combing steps after the breaking card (CaCoD) lead to a decrease in the quantity of small diameter fibres (below 50 μ m). This is accompanied by an increase in the quantity of fibres of intermediate diameters (between 50 and 200 μ m).

The increase in the average diameter of the bundles in CaCoD compared to those in ScD and CaD could therefore be explained in part by a partial disappearance of the smaller fibres, during the passage of the material through the finishing card and tow-combing machine.

3.2. Comparison of fibre bundle lengths

The length of fibres at the end of the various extraction/processing stages is an important parameter to control for traditional spinning. For composite application, it is also better to work with as narrow as possible fibre length distribution so that to obtain as homogeneous as possible yarns.

The results obtained and presented in Fig. 7 show, a significant reduction in the median length of the fibres (weighted by the length) between Sc and ScD, from 145 mm to 90 mm. A significant reduction in the length of carded hemp bundles was also observed over the course of the treatments (CaD and CaCoD) compared to the carded reference material (Ca). Regardless of the extraction route used, drawing thus leads to a significant decrease in the mean of fibre lengths (ScD VS Sc and CaD VS Ca) (Fig. 7). However, the materials after drawing (ScD and CaD) do not show significant differences in terms of length. As mentioned above, the material obtained from CaD is less suitable to manufacture composite materials due to the impurities. Additional processing steps were therefore used. The results obtained (CaCoD) show that, logically, the additional carding and combing steps lead to a significant decrease in the lengths of the bundles compared to the material of ScD but no significant differences with CaD. No significant decrease in fibre diameter was either observed between CaD and CaCoD. This length is sufficient however for the manufacture of roving type varns.

A more detailed study of the fibre length distributions in each of the batches shows a clear difference between the population of the scutched/hackled samples and those of the carded route. In scutched and hackled fibres (ScD) long fibres (more than 20 cm) remain even though fibres of less than 15 cm are the most represented (Fig. 9). On the



Fig. 6. Diameter diagrams of the fibre bundles of the different batches (grey bars and dotted line represent the response of the batches taken together).



Fig. 7. Fibre lengths distributions of the fibre bundles weighted by the length; letters indicate the groups with no statistical differences.

contrary, carding (CaD and CaCoD) leads to a greater breakage of the fibres and therefore to the disappearance of these long fibres. The increase in the number of extraction stages in CaCoD leads to an even greater representation of medium and small fibres and to the complete disappearance of long fibres (more than 20 cm). This is interesting as the fibre length becomes more homogeneous and therefore favours a further homogenization during the supplementary drawing and roving steps which take place to manufacture roving type yarns. The fibre length distribution becomes also more homogeneous in CaCoD and this may be interesting in terms of composite property distribution (Fig. 8).

3.3. Mechanical properties of the elementary fibres

To obtain a favourable transfer of load to fibres, it is important to get a good separation of the technical fibres as described above. It is also important to keep the intrinsic tensile potential of the fibres as high as possible.

The results obtained show, a reduction in the average stresses at break between Sc and ScD without statistical difference. The same trend was observed between Ca and CaD. CaCoD showed the highest stresses at break statistically different from Sc and ScD. The elastic moduli vary little between the different batches, although Ca shows the highest average elastic modulus. No statistical difference is observed between



Fig. 8. Fibre length distributions in each batch (grey bars and dotted line represent the response of the batches taken together).



Fig. 9. Individual fibre elastic modulus (A) and strength (B); letters indicate the groups with no statistical differences.

the batches.

The mechanical tensile tests on single elementary fibres (Fig. 9) show, that drawing (Sc VS ScD and Ca VS CaD) does not have a significant influence on the mechanical properties of individual fibres, either in terms of modulus or strength at failure, regardless of the type of extraction. Thus, this process, which is necessary for the use of the fibres in traditional spinning, provides a significant improvement in the fibres bundle division while maintaining mechanical properties similar to what was obtained before drawing. These results can be correlated with observations made recently by [23] who observed that higher mechanical properties are obtained after high-intensity hackling than if softer treatment parameters are used. This behaviour is explained by a reduction in the surface area of the kink-bands present in the fibres. It is also accompanied by a change in stress strain curve type (from type 2, 3 (non linear) to type 1 (linear)) attributed to a local reorientation of the cellulose micro-fibrils and to pre-stretching. The same explanations can be given to explain the low impact of drawing.

The materials obtained after drawing for the two extraction routes (ScD and CaD) also do not show significant difference, as presented in Fig. 9 with same letter for the statistical test, in mechanical properties (stress at break and elastic modulus). This is consistent with what was observed for the morphological properties of the fibres (fibre lengths and diameters). Thus, the all-fibre extraction route using breaking roller/breaking card leads to fibres with similar mechanical and morphological properties to those obtained by the scutching/hackling route, while reducing production costs. This is particularly true at the harvesting and dew retting steps during which classical equipment for hay can be used instead of very costly and specific harvesting and retting management machines (especially at the harvesting step). In addition, the mechanical properties obtained are higher to those of hemp fibres extracted using a hammer mill device (285 MPa and 14 GPa for strength and modulus respectively) [12,24] and closed to those obtained for textile flax fibres (59.8GPa and 527 MPa) [22]. It is also interesting to note that the hemp fibres obtained in CaD and CaCoD were processed using equipment that can be implemented at a lower price than traditional scutching and hackling plants that are at the moment only encountered in the traditional textile flax zone.

As explained above, the material obtained after carding (CaD) still contains small pieces of shives (less than 1 % in mass) which may have

an impact on the mechanical properties of the composites. Additional processing steps (finishing carding and rotational combing) was therefore tested after carding and before drawing (CaCoD). This also did not significantly affect the strengths and elastic moduli of the fibres, either in comparison to the carded and drawn fibres only (CaD) or to the material obtained by scutching/hackling (ScD). As the fibres obtained after combing (CaCoD) are clean of any waste and are not mechanically degraded, it might therefore be preferable to favour the latter extraction technique to provide cleaner and more homogeneous fibres for obtaining high potential composite materials.

To investigate if the tensile potential of the fibres can be correlated to a composite reinforcement level, UD composite coupons were manufactured and tested from the fibres of ScD, CaD and CaCoD in order to quantify the impact of the various treatments on the finished material.

3.4. IFBT results

A non-linear response is obtained in the tensile tests of the IFBT specimens, Fig. 10. This behaviour can be compared to that obtained on individual fibres for a type II curve. It shows non-linearities. It is first linear up to a change of slope which shows a decrease in stiffness.

Fig. 11 shows the evolution of the back-calculated breaking strengths of the fibres within the composites after drawing for each extraction route (ScD, CaD and CaCoD). Statistical analyses indicate that there are no differences between ScD and CaCoD. There is only a statistical difference between CaD and CaCoD. This can be explained by the fact that CaD still contains small pieces of shives that may be considered as defects in the composite structure and a place of crack initiation. The fibres strengths depend on the defects, surface defect, kink-bands within the fibres [25,26]. The stress concentrations that can occur in these critical areas not only weaken the fibre but can also influence the composites by decreasing their mechanical properties through the initiation of micro cracks in the matrix [27]. More recently, Quereilhac et al. [28] showed that kink-band zones are places of intense stress concentration (up to 8 times the stress in non-defect zone). So, carding which is an aggressive process may be the place of kink-band defect generation. However, the greater number of extraction steps leading to CaCoD in comparison to CaD does not lead to lower back-calculated fibre strength as was observed for direct test on elementary fibres. The rotational combing







Fig. 11. Back-calculated fibre elastic modulus (A) and strength (B); letters indicate the groups with no statistical differences.

after carding has for effect to remove the last remaining pieces of shives and they are therefore useful to increase the composite reinforcement potential of the fibres. It is also expected that this process has for effect to provide tensile loads to the fibres and partially remove or decrease the severity of kink-band structural defects.

The mechanical properties of the IFBT specimens are close to or even better (increase of more than 50 %) in terms of stresses at break than those obtained in [29]. In this study, composite materials were manufactured from hemp fibres extracted from disordered lines. The tensile and flexural properties obtained were shown to be suitable for use in components subject to high loads. This work confirms the high reinforcement potential of carded fibres and their possible use in structural composites.

The elastic moduli of the fibres do not, however, vary significantly between the batches of fibres from the different extraction routes (Fig. 11 (A)). Beyond the presence of kink-band defects, the elastic moduli are impacted by structural changes at the local scale of these defects. Thus, the sudden increase in the angle of the cellulose microfibrils in the kink-bands [30] leads to a decrease in the elastic moduli of the fibres [31] without having a significant impact on their strengths at break [32–34]. Similarly, the presence of pores [35] within kink bands should contribute to a reduction in the fibre stiffness [30]. This effect may not be significant if the defect density is not too high but will be exponentially inverse with fibre elastic moduli. Like for the elementary fibres, no statistical difference is observed between ScD, CaD and CaCoD back-calculated moduli. Even if more processes and therefore more chances to create kink-band defects may take place for ScD and CaCoD than in CaD, compensation effects such as the ones described above with possible microfibril reorientation and pore closure in kink-band may take place during combing and therefore moderate the influence of kinkband defects as already observed by [9] in a study dealing with scutched and hackled hemp fibres.

The results presented in the previous sections demonstrate that the carded route approach can provide high performance fibres, equivalent to the ones produced using the classical scutching and hackling route (ScD). In our results, the less refined fibres (CaD) lead to composite reinforcement potential lower than the one which possess shive free fibres (CaCoD) and ScD fibres which also do not exhibit any shives. It is first surprising to observe that fibres processed using a multiple carding approach which was expected to be more aggressive (CaCoD) show similar properties to the ones of scutched and hackled fibres. This is probably due to the final combing and drawing that are in common at the end of the 2 depicted approaches (ScD and CaCoD). These process may tend to homogenise the number and severity of structural defects and create fibres with more linear tensile behaviour as shown in [36]. This therefore means that it is interesting to process the fibres with extra finishing card and combing after the breaking card stage on a mechanical point of view and also to obtain more homogeneous slivers and roving type yarns even if the mechanical properties of the fibres within the roving are not studied in this work. As the roving process consists mostly of drawing and twisting, it is expected that the properties of fibres are not changed or may be increased for the strength (if one expects a reduction of the kink-band defect size) in comparison to ScD or CaCoD.

4. Conclusions and perspectives

Hemp fibres processed using an all-fibre extraction route based on breaking roller/breaking card approach were transformed into roving type yarn and characterised to investigate their potential as a reinforcement material for load bearing composite materials. Both morphological and mechanical properties were investigated and compared to fibres coming from the same batch but extracted using a more classical scutching and hackling approach. Results showed that the fibres extracted using the carded route approach show equivalent level of divisibility and tensile properties than for classical reference approach (scutching/hackling). This therefore constitutes a very important step towards the possibility to use alternative approaches to scutching and hackling facilities which are encountered in textile flax territory. Using such devices could open the possibility to new territories to produce hemp fibre grades suitable for a use in load bearing composites. This work also demonstrates that the hemp used in this work, produced in Northern Italy was sufficiently dew retted. This confirms the fact that hemp can grow in many places in Europe and can be also dew retted in different territories than the one dedicated traditionally to textile flax and nowadays hemp. Opening such new territories for hemp fibre production for technical load bearing applications would be very beneficial for large scale industries such as automotive. These industries are, at the present time, looking for larger natural fibre quantities from Europe and that cannot be produced at the present time in traditional flax production zone because almost all the fibres produced in this zone are dedicated to garment textile needs which is in always increasing demand of more sustainable fibres.

CRediT authorship contribution statement

Marie Grégoire: Writing - review & editing, Writing - original draft,

Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Mahadev Bar: Writing - review & editing, Investigation. Xavier Gabrion: Writing - review & editing, Data curation. Gilles Koolen: Writing - review & editing, Data curation. Salvatore Musio: Writing - review & editing, Investigation, Data curation. Debora Botturi: Writing - review & editing, Investigation. Giorgio Rondi: Writing - review & editing, Resources, Conceptualization. Stefano Amaducci: Writing - review & editing, Supervision, Methodology, Investigation, Conceptualization. Emmanuel De Luycker: Writing review & editing, Visualization, Validation, Supervision, Methodology, Formal analysis, Data curation. Aart Van Vuure: Writing - review & editing, Supervision, Conceptualization. Vincent Placet: Writing - review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. Pierre Ouagne: Writing - review & editing, Writing - original draft, Supervision, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The research leading to these results has received funding from the European Union Horizon 2020 Framework Program for research and innovation under grant agreement no. 744349.

The authors would also like to thank the "Terre de Lin", "Linificio e Canapificio" and Hemp-Act companies for opening their fibre extraction and fibre preparation industrial facilities as well as some of their characterization equipment.

For the purpose of Open Access, a CC-BY public copyright licence has been applied.



Data availability

Data will be made available on request.

References

- Carus M, Karst S, Hobson J, Bertucelli S. The European Hemp Industry: Cultivation, processing and applications for fibres, shivs and seeds. Eur Ind Hemp Assoc 2017; 1994:1–9. https://doi.org/10.1016/j.jallcom.2015.04.081.
- [2] Fitzgerald A, Proud W, Kandemir A, Murphy RJ, Jesson DA, Trask RS, et al. A life cycle engineering perspective on biocomposites as a solution for a sustainable recovery. Sustain 2021;13:1–25. https://doi.org/10.3390/su13031160.
- [3] Kandemir A, Longana ML, Panzera TH, Del Pino GG, Hamerton I, Eichhorn SJ. Natural fibres as a sustainable reinforcement constituent in aligned discontinuous polymer composites produced by the HiPerDiF method. Materials 2021;14(8): 1885. https://doi.org/10.3390/ma14081885.
- [4] Amaducci S, Scordia D, Liu FH, Zhang Q, Guo H, Testa G, et al. Key cultivation techniques for hemp in Europe and China. Ind Crops Prod 2015;68:2–16. https:// doi.org/10.1016/j.indcrop.2014.06.041.
- [5] Bouloc P, Allegret S, Arnaud L, editors. Hemp: industrial production and uses. CABI; 2013.
- [6] Höppner F, Menge-Hartmann U. Yield and quality of fibre and oil of fourteen hemp cultivars in Northern Germany at two harvest dates. Landbauforsch Volkenrode 2007;57:219–32.
- [7] Grégoire M, Bar M, De Luycker E, Musio S, Amaducci S, Gabrion X, et al. Comparing flax and hemp fibres yield and mechanical properties after scutching/ hackling processing. Ind Crops Prod 2021;172. https://doi.org/10.1016/j. indcrop.2021.114045.
- [8] Horne MRL, Waldron D, Harwood JL, Harwood RJ. The production and extraction of flax-fibres for textile fibres. J Biobased Mater Bioenergy 2010;4:98–105.
- [9] Gabrion X, Koolen G, Grégoire M, Musio S, Bar M, Botturi D, et al. Influence of industrial processing parameters on the effective properties of long aligned European hemp fibres in composite materials. Compos Part A Appl Sci Manuf 2022; 157:106915. https://doi.org/10.1016/j.compositesa.2022.106915.
- [10] Müssig J. Industrial Applications of Natural Fibres. John Wiley; 2010.

- [11] Bourmaud A, Beaugrand J, Shah DU, Placet V, Baley C. Towards the design of highperformance plant fibre composites. Prog Mater Sci 2018;97:347–408. https://doi. org/10.1016/j.pmatsci.2018.05.005.
- [12] Placet V, Trivaudey F, Cisse O, Gucheret-Retel V, Boubakar ML. Diameter dependence of the apparent tensile modulus of hemp fibres: A morphological, structural or ultrastructural effect? Compos Part A Appl Sci Manuf 2012;43: 275–87. https://doi.org/10.1016/j.compositesa.2011.10.019.
- [13] Pinsard L, Revol N, Pomikal H, De Luycker E, Ouagne P. Production of Long Hemp Fibers Using the Flax Value Chain. Fibers 2023;11:1–17. https://doi.org/10.3390/ fib11050038.
- [14] Graupner N, Lehmann KH, Weber DE, Hilgers HW, Bell EG, Walenta I, et al. Novel low-twist bast fibre yarns from flax tow for high-performance composite applications. Materials (Basel) 2021;14:1–27. https://doi.org/10.3390/ ma14010105.
- [15] Bar M, Grégoire M, Kahn SU, De Luycker E, Ouagne P. Study on classically harvested linseed flax fibers for bio-composite reinforcement and textile applications. J Nat Fibers 2022. https://doi.org/10.1080/ 15440478.2021.2024934.
- [16] Coroller G, Lefeuvre A, Le Duigou A, Bourmaud A, Ausias G, Gaudry T, et al. Effect of flax fibres individualisation on tensile failure of flax/epoxy unidirectional composite. Compos Part A Appl Sci Manuf 2013;51:62–70. https://doi.org/ 10.1016/j.compositesa.2013.03.018.
- [17] Sisti L, Totaro G, Vannini M, Celli A. Retting process as a pretreatment of natural fibers for the development of polymer composites. Lignocellulosic composite materials 2018:97–135. https://doi.org/10.1007/978-3-319-68696-7_2.
- [18] Garat W, Corn S, Le Moigne N, Beaugrand J, Bergeret A. Analysis of the morphometric variations in natural fibres by automated laser scanning: Towards an efficient and reliable assessment of the cross-sectional area. Compos Part A Appl Sci Manuf 2018;108:114–23. https://doi.org/10.1016/j.compositesa.2018.02.018.
- [19] Grégoire M, De Luycker E, Ouagne P. Elementary liber fibres characterisation: bias from the noncylindricity and morphological evolution along the fibre. Fibers 2023. https://doi.org/10.3390/fib11050045.
- [20] ISO 10618:2004. Carbon fiber Determination of tensile properties of resinimpregnated yarn. Int Stand Organ 2005.
- [21] CELC. Impregnated Fibre Bundle Test IFBT Methodology of uses. 2015.
 [22] Bensadoun F, Verpoest I, Baets J, Müssig J, Graupner N, Davies P, et al. Impregnated fibre bundle test for natural fibres used in composites. J Reinf Plast Compos 2017;36:942–57. https://doi.org/10.1177/0731684417695461.
- [23] Morgillo L, Melelli A, Ouagne P, Scheel M. Industrial Crops & Products Elucidating links between the mechanical performance of flax fibres and their structural defects. Ind Crop Prod 2023;206. https://doi.org/10.1016/j.indcrop.2023.117722.
- [24] Placet V. Characterization of the thermo-mechanical behaviour of Hemp fibres intended for the manufacturing of high performance composites. Compos Part A

Appl Sci Manuf 2009;40:1111–8. https://doi.org/10.1016/J. COMPOSITESA.2009.04.031.

- [25] Richely E, Bourmaud A, Placet V, Guessasma S, Beaugrand J. A critical review of the ultrastructure, mechanics and modelling of flax fibres and their defects. Prog Mater Sci 2022;124:100851. https://doi.org/10.1016/j.pmatsci.2021.100851.
- [26] Hughes M. Defects in natural fibres: Their origin, characteristics and implications for natural fibre-reinforced composites. J Mater Sci 2012;47:599–609. https://doi. org/10.1007/s10853-011-6025-3.
- [27] Greenwood J, Rose P. Compressive behaviour of Kevlar 49 fibres and composites. J Mater Sci 1974;9:1809–14.
- [28] Quereilhac D, De Luycker E, Guessasma S, Abida M, Perrin J, Weitkamp T, et al. Synchrotron X-ray microtomography and finite element modelling to uncover flax fibre defect's role in tensile performances. Compos Part A 2024;184:108276. https://doi.org/10.1016/j.compositesa.2024.108276.
- [29] Graupner N, Weber DE, Bell EG, Lehmann KH, Hilgers HW, Randerath H, et al. Hemp From Disordered Lines for New Staple Fibre Yarns and High-Performance Composite Applications. Front Mater 2022;8:1–20. https://doi.org/10.3389/ fmats.2021.807004.
- [30] Melelli A, Durand S, Arnould O, Richely E, Guessasma S, Jamme F, et al. Extensive investigation of the ultrastructure of kink-bands in flax fibres. Ind Crops Prod 2021; 164. https://doi.org/10.1016/j.indcrop.2021.113368.
- [31] Davies GC, Bruce DM. Effect of Environmental Relative Humidity and Damage on the Tensile Properties of Flax and Nettle Fibers. Text Res J 1998;68:623–9. https:// doi.org/10.1177/004051759806800901.
- [32] Andersons J, Poriķe E, Spārniņš E. Modeling strength scatter of elementary flax fibers: The effect of mechanical damage and geometrical characteristics. Compos Part A Appl Sci Manuf 2011;42:543–9. https://doi.org/10.1016/j. compositesa.2011.01.013.
- [33] Baley C. Influence of kink bands on the tensile strength of flax fibers. J Mater Sci 2004;39:331–4. https://doi.org/10.1023/B:JMSC.0000007768.63055.ae.
- [34] Thygesen LG, Eder M, Burgert I. Dislocations in single hemp fibres-investigations into the relationship of structural distortions and tensile properties at the cell wall level. J Mater Sci 2007;42:558–64. https://doi.org/10.1007/s10853-006-1113-5.
- [35] Quereilhac D, Pinsard L, Guillou E, Fazzini M, De Luycker E, Bourmaud A, et al. Industrial Crops & Products Exploiting synchrotron X-ray tomography for a novel insight into flax-fibre defects ultrastructure. Ind Crop Prod 2023;198:116655. https://doi.org/10.1016/j.indcrop.2023.116655.
- [36] Guillou E, Bar M, Scheel M, Falher T, Weitkamp T, Shah DU, et al. Use of a commingling process for innovative flax fibre reinforced unidirectional composites. Compos Part B Eng 2024;270:111150. https://doi.org/10.1016/J. COMPOSITESB.2023.111150.