Influence of industrial processing parameters on the effective properties of long aligned European hemp fibres in composite materials

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Abstract

Hemp is a sustainable source of natural fibres that can contribute to meet the increasing demand for technical applications in the textile and the composite sectors. Continuous reinforcements can be produced using the existing flax machinery, initially developed for textile purposes. To achieve competitive and economically viable fibre yields and a fibre quality suitable for secondary processing and composite application, hemp needs to be adequately selected and prepared and the flax machinery and settings have to be adapted to the hemp specificities. In this context, this paper studies the influence of agronomic features and processing stages and settings on the effective tensile properties of fibres extracted from two hemp varieties determined using impregnated fibre bundle tests. Results show that the effective properties of fibres are maintained and even improved during processing, in particular during the hackling and stretching steps. Hemp can achieve properties comparable to high quality long flax fibres.

Keywords: A. Natural fibers; A. Biocomposites; B. Mechanical properties

1 Introduction

Hemp (Cannabis sativa L.) is a multiuse, multifunctional crop that provides raw material to a large number of traditional and innovative industrial applications. Traditionally, its main product was the long bast fibre; now it is cultivated as a dual-purpose crop, for the fibre and the seed [1], or as a multipurpose crop when also the flowers of threshing residues are used to extract high value cannabinoids [2].

The use of hemp bast fibre, traditionally linked to the production of textiles, ropes, twines and paper pulp, is now considered for the production of insulation materials or to reinforce composites. Hemp fibres during processing are separated from the woody core (shives), a by-product that has a wide range of applications from the production of MDF to bio-building material, even though its main application is for animal bedding.

Hemp, which is well adapted to a wide range of environments, is cultivated all over the globe and its acreage is increasing in China, Europe and North America.

Hemp is an environmentally friendly and fast-growing annual crop. It is thereby a substantial consumer of carbon dioxide with an absorption of approximately 1.4 to 1.6 t of CO_2 per tonne of hemp [3, 4]. With a yield average of 5.5 to 8 t ha^{-1}, this represents 9 to 13 tonnes of CO_2 absorption per hectare harvested [5]. In that respect, hemp provides a carbon-negative material for engineering. Hemp also requires limited amount of water to be produced [6]. Due to its vigorous growth, shading capacity and disease resistance, hemp can be grown without the use of herbicide, pesticide or fungicide. It also regenerates and improves the quality of soils. Inputs of fertilisers are low [7] and the interventions and manpower requirements for farming are limited. The resulting energy cost for raw hemp fibre production is estimated at approximately 5 GJ t^{-1}, about 7 times less than for glass fibres [3]. Expressed in CO_2 equivalents, the approximate production cost is only 680 kg eq. CO_2 t^{-1} for hemp fibres in comparison to the 2500 and 4000 kg eq.CO_2 t^{-1} required for example for the production of respectively PP and PET fibres [3]. Thus, it constitutes an interesting alternative to mineral and synthetic fibres.

In Europe, hemp is currently processed using mechanical systems, based on beating (hammer mills) and or fast rotating nailed rollers (referred to as decorticators), which provide fibres in the form of short and medium-length fibres from disordered straws [8]. These very efficient but also very aggressive processing methods are very damaging for the fibres as very high loads are transferred to them. As a consequence, a high number of defects such as kink-bands is observed and this number increases as a function of the process severity [9]. The mean values of their resulting tensile properties are generally significantly lower than for textile flax [10-12]. An alternative to these processing routes (hammer mills or decorticators) is to use aligned straws and the scutching and hackling machinery dedicated to textile flax [8]. Several authors have investigated the production of long hemp processed with such machinery in view of textile and high-added value applications [13-15]. Musio et al. [13] demonstrated that using such flax machinery and a well-controlled retting, hemp can achieve properties comparable to high quality long flax fibres for high performance composites, with tensile stiffness and strength reaching more than 55 GPa and 450 MPa, respectively, to be compared to 59.8 GPa and 527 MPa measured for industrially hackled flax [16]. In this study, they obtained quite low scutching yields of long-aligned fibres and high amounts of scutching tows. Grégoire et al. [17] demonstrated recently, at a laboratory scale, that the process parameters can be tuned to significantly improve the long line hackled fibre yields up to 18% of the straw mass and thus obtain values competitive to the flax ones if one considers that hemp shows generally a larger stem yield than flax. Vandepitte et al. [14] also used industrial flax scutching facilities with some of the process parameters changed for hemp extraction purposes with a wide range of European hemp varieties. When compared to Musio et al. [13], higher levels of long fibre scutching yields were obtained but these ones were dependent on the batches, varieties and levels of dew retting. So, there is a great interest and need for optimising the industrial processing of hemp using the scutching and hackling flax machinery. The optimisation work is dedicated to the scutching process settings and not to any change of the different tools such as breaking rollers or beating turbines. Of course, this question would be interesting to raise as the hemp stem diameters are globally larger than the flax ones, especially when the hemp is grown for biomass purpose. When hemp is grown for fibre purpose and especially for garment textile or load bearing composite with fibre extraction performed using scutching equipment, the hemp fibre stem diameters may be considerably reduced to levels lower than 5 mm by increasing the plant density. This is still higher than the flax stem diameter and the flax breaking rollers are probably not perfect, but at the present time, it is not possible to change the design of industrial scutching plants as these process hemp only for experiments or small-scale productions and textile flax the very vast majority of the time.

The question of the fibre properties to be considered for such optimisation can be raised. Indeed, for textile applications, fibre tenacity, fineness, cleanness and colour are often selected. For composite applications, the choice of designers is mainly driven by the fibres' strength and rigidity (stiffness). As of today, there are mainly three different experimental methods to determine the tensile properties of fibres: (i) single fibre tensile test (SFTT), (ii) dry fibre bundle test (FBTT) and (iii) Impregnated Fibre Bundle Test (IFBT).

SFTT is the most widely applied method for the measurement of the tensile properties of synthetic fibres. For plant fibres, the test is more challenging and time-consuming. For flax, the test is standardized (NFT25-501-2 and NFT25-501-3). The accuracy of the measurement is directly dependent on the fibre preparation (extraction, handling), the experimental settings (fibre alignment and clamping, gauge length, strain rate, hygrothermal conditioning…), data collection (measurement of load and displacement or strain) and post-processing (machine compliance correction, determination of stress and modulus, loading history, assumptions related to isotropy and homogeneity…) [18, 19]. For the strength and stiffness, the most influential parameter, generally leading to large error and scattering in the measurements, is the determination of the cross-sectional area of the fibre [20-27]. A large quantity of fibres (from at least 50 to few hundreds) has to be tested to ensure a reliable analysis of results. A source of confusion and uncertainty also comes from the fact that SFTT can be applied at the scale of individual (elementary) fibres and fibre bundles ('technical' fibres). These two cases are not always distinguished and the impact of the pectic interface on the measured properties can be significant. For the design of composites, the nature of the tensile properties to be used can also be questioned since, whatever the type of continuous reinforcement used, the resulting composite is reinforced by both individual fibres and bundles of fibres.

In FBTT [28], a collection of several fibres are connected in parallel with both ends clamped to a support. While quite easy to implement for synthetic fibres which are produced in the form of continuous monofilaments, this test is very challenging for plant fibres due to their finite length and the resulting discontinuities within rovings and yarns.

The IFBT is a well-established method for carbon and glass fibres, standardised for continuous and staple-carbon fibre yarns (ISO 10618:2004). It was also adapted a few years ago for natural fibres and validated by a round robin exercise on flax fibres [16]. In this method, an unidirectional composite is manufactured and loaded in the fibre direction. The fibre stiffness and strength are then identified by inverse method from the measured composite properties and a micromechanical model, generally the rule of mixtures. A good impregnation quality with a negligible content of residual voids is required [29]. If initially conceived and used for tensile loading, its use under compressive loading has also been investigated more recently for flax [30].

Literature pointed out that the properties measured using these different testing methods can be significantly different [31-34]. Shah et al. [32] explored the potential sources of the observed discrepancy and concluded that the more likely origins relate to both measurement uncertainties and inaccuracy in predictions based on the rule-of-mixtures. However, the main advantages of IFBT lie in the simplicity of the preparation of the specimens and in the implementation of the test and also in the fact that a large quantity of fibres is tested simultaneously, including individual fibres and bundles of fibres. It also gives access to the effective properties of the fibres, i.e. the reinforcing potential of the fibre in the matrix, resulting from the fibre properties but also from the fibre-matrix interfacial bonding, the fibre individualisation and spatial distribution in the resin. So, it is considered so far as the most efficient method to determine quickly and reliably the effective properties of fibres as they behave in the composite.

The objective of this study is to provide pieces of knowledge for the optimisation of the industrial processing of aligned hemp straws using the scutching and hackling flax machinery. The influence of the processing stages (including scutching, hackling, sliver forming, doubling and stretching) and settings on the effective properties of fibres is investigated on two hemp (*Cannabis sativa L.*) varieties, namely Futura 75 and Fibror 79 cultivated in Italy in the frame of the BBI-JU project SSUCHY (www.ssuchy.eu). Hemp is a widespread crop, well adapted to a broad range of environmental conditions. Traditionally Italy was the European leader in the production of fibre hemp with up to

130.000 ha of cultivations and still now most of the European varieties have been bred from Italian genotypes. The two selected varieties are both bred in France by Hemp-It and are relatively similar for their cycle, habitus and productivity and they are both well adapted to Italian conditions. Interestingly, Fibror 79 is a « yellow » variety, which is easier to process than traditional ones [35].

Tests were realised on hemp straws harvested and retted in 2018 and 2019. The sowing and harvest times as well as the stem portion are considered in the analysis. The mechanical properties of the long-aligned fibres obtained for the different batches at the different stages of the processing are evaluated using IFBT.

2 Materials and methods

2.1 Materials and processing

2.1.1 Hemp stems: field trials, varieties and harvesting

Hemp straws were obtained from large-scale field trials carried out in Piacenza, Italy (45° 3' 9.436" N 9° 41' 34.742" E) in 2018 and 2019 with two monoecious varieties, a green one Futura 75 (FUT) and a yellow one Fibror 79 (FIB). They were sowed with a target plant density of 150 plants m^{-2}. The field was fertilized with 60 kg of nitrogen per ha.

In 2018, the sowing was carried out on 25th April with a seed rate of 50 kg/ha. Stems were harvested at two times: at the end of flowering on 11th August, and at seed maturity on 28th September as a dual valorisation increases the income of the farmer (H2). Stems from H1 were dew-retted for 5 weeks (until 18th September), which was judged as appropriate for a good retting level according to the colour of the stems.

In 2019, a similar protocol than in 2018 was followed with an early sowing time in April and two harvesting times (H1 early august) and H2 (early September). Stems were left on the field until mid-October to obtain a complete and optimum level of retting.

For the harvest, a prototype machine was used. It enabled an efficient cut of the stems and the formation of swaths of aligned stems. The harvested plants were then laid in the field for the dew-retting. A second prototype was used to cut the plants in 1-meter stem portions and to turn the swath to improve retting homogeneity. The bottom and middle part of the plant, cut in 1-meter portion, were baled separately. The meteorological data (rainfall and temperature) are added in the supplementary information.

2.1.2 Fibre processing

Figure 1: Schematic representation of the processing stages and resulting products

Long aligned fibres were extracted using industrial flax machinery, specifically, an industrial Depoortere scutching device and a Linimpianti hackling machine located at the Terre de Lin company (Saint-Pierre-Le-Viger, France). The scutching machine is composed of two distinct devices: a breaking system composed of a succession of horizontal fluted rollers and a beating stage which consists of successive pairs of rotating turbines, with each turbine rotating in opposite direction. For the hemp straw harvested in 2019, two different settings were used for the scutching, breaking and beating steps, labelled R1 and R2. R1 corresponds to the high speed (settings used for flax processing) while R2 corresponds to a lower speed. The exact values of speed and settings are confidential and cannot be given here.

At the end of the hackling line a continuous sliver with a large count (linear mass of about 15,000 tex) was realised. This sliver was then processed into rovings at an industrial scale in Linificio e Canapificio Nazionale (Villa d'Almè, Italy). The sliver was drawn and doubled several times and slightly twisted to obtain at the end a roving of 350 Tex with a twist level of approximately 35 turn/meter. This process was also performed at the lab scale drawing system from Linimpianti company (called Mini-Sytem), usually used in the industrial flax processing to evaluate the spinning ability of scutched and hackled fibres. This was used in this study to evaluate if any difference in the effective properties of the fibres can be detected between the matters processed with the Mini-System (supposed to mimic the industrial process) and the industrial process itself. Indeed, the Mini-system simulates, at a reduced scale, the six drawing/doubling stages used in the flax spinning industry to prepare the slivers into rovings that will be used at the spinning stage. It consists of six drawing stages during which the linear mass of the sliver is decreased up to 150 Tex. During the different stages of this process, six parallel “Gill type” systems perform the different drawing operations. During this stage, the sliver mass is reduced but it is also homogenised as between each drawing stage, six drawn slivers are each time grouped together before the following drawing. During these operations, the technical fibre diameter may also be reduced when the technical fibres are pulled from the Gill system pins.

The whole process is schematically represented in Figure 1. To facilitate the identification of the matter, the following label will be used (See Figure 2). The label fields and entries are described in Table 1.

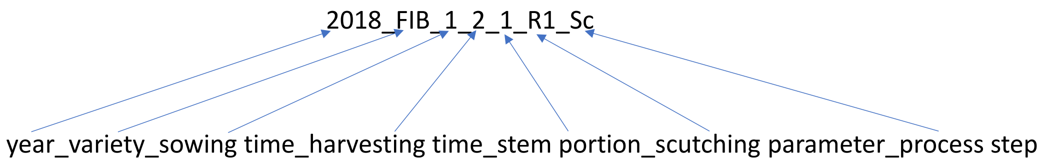

Figure 2. Identification of the matter.

Table 1: Label fields and entries used for the nomenclature of the fibre samples

Label fields	**Label entries**				
Year	2018	2019			
Variety (VAR)	FUT = Futura 75	FIB – Fibror 79			
Sowing time (S)	1 = first sowing time	2 = second sowing time			
Harvesting (H)	1 = Full flowering	2 = seed maturity			
Stem portion	1 = 1st meter	2 = 2nd meter			

Scutching parameter settings	R1 = flax settings	R2 = reduced speed			
Process stage	Sc = scutching	Ha = hackling	Sl = sliver forming	MS = doubling/stretching using MiniSystem	R = doubling/stretching using industrial equipment

The production of continuous reinforcement from the discontinuous hemp fibres requires a certain minimum quantity. This quantity has not been systematically reached for all the batches. Therefore, for the two tested years (2018 and 2019), some of the batches produced the same year were mixed together to form a single sliver or roving. They were labelled 2018_MIX_Sl, 2018_MIX_MS, 2018_MIX_R and 2019_MIX_R, respectively.

2.2 IFBT specimens manufacturing

IFBT specimens were prepared following the technical document "Impregnated Fibre Bundle Test – IFBT – Methodology of uses" published by the European Confederation of Flax and Hemp (CELC) [36]. This work being carried out within the framework of a European collaborative project, the specimens were prepared in two different laboratories, labelled A and B, using the same above-mentioned protocol and some small adjustments related to the know-how and previous experiences of the involved research teams. The exact protocols are described below. For both, the IFBT specimens were manufactured by aligning the long fibres obtained at the different stages of the processing and impregnating them with an epoxy system.

Protocol A (UFC)

The specimens were polymerized from the GreenPoxy 56 resin and the SD 7561 hardener provided by Sicomin company. The fibre samples were placed in the mould cavity after being conditioned at a temperature of 23°C and a relative humidity of 50% during few hours to reach equilibrium. The resin was poured on top of the fibres as the fibres were placed in the mould. The quantity of resin used was calculated to reach a fibre volume fraction target in composites of approximately 40%. The counter-mould was then placed on the top. No spacer was used to limit the porosity level. The specimens were then cured at 60°C during 1h under a pressure of 2 bars and demoulded. A post-curing at 130°C was then realised during 1h. The dimensions of the manufactured specimens were approximately 200 mm x 16 mm x 1 mm. The manufactured IFBT specimens were then stored in a climatic chamber at 23°C and 50% RH during a minimum of four weeks to reach the equilibrium moisture content. The dimensions and mass of each specimen were measured. At least, six specimens were manufactured for each tested condition.

The fibre volume fraction V_f, matrix volume fraction V_m and void content V_v were determined using the following equations:

$$V_f = W_f \times \frac{\rho_c}{\rho_f} \quad (1)$$

$$V_m = (1 - W_f) \times \frac{\rho_c}{\rho_m} \quad (2)$$

$$V_v = 1 - V_f - V_m \quad (3)$$

where W_f is fibre weight fraction determined as the ratio of the measured mass of fibres to the measured mass of composite, ρ_c, ρ_f and ρ_m specific gravity of composite, fibre and matrix, respectively. ρ_c was determined as the ratio of the measured mass of composite to the measured volume of composite and ρ_f and ρ_m were previously determined by pycnometry with values equal to 1.503 g/cm^3 and 1.17g/cm^3, respectively.

Protocol B (KU Leuven)

Protocol B is similar to A except for the following points. An Epikote 828 LVEL/ Dytek DCH-99 epoxy system was used. The fibres were dried during at least 24h at 60°C before the manufacturing of the IFBT specimens. The specimens were cured at 150°C during 2h in a manual hydraulic press. Spacers were used in the mould to approach a specimen thickness of 2mm. They were then conditioned for at least one month at 21°C and 54% RH above a salt solution. The dimensions of the manufactured specimens were approximately 200 mm x 10 mm with a thickness varying between 1.6 and 2 mm.

2.3 Testing methods

2.3.1 Fibre fineness

The fineness was measured using a FiberShape device developed by IST AG (Vilters, Switzerland). It consists of a high-precision Reflecta MF 5000 scanner (Reflecta, Eutingen im Gaü, Germany), associated with the Silverfast fibre recognition software developed by Lasersoft Imaging (LaserSoft Imaging, Kiel, Germany).

No particular pre-treatment (temperature or humidity stabilization, etc.) was carried out on the fibres. The technical fibres were only cut to a fixed length before being fed into the scanner.

The parameters used are as follows: bundle length: 2cm; measurement accuracy: 3200 dpi; number of measurements: 4 000-10 000. This number, corresponding to the number of scanned technical fibres is necessary to establish a good representation of the fibrous population. The number of scans depends on the number of fibres placed on the scanner.

2.3.2 IFBT

Tensile tests were done on the produced IFBT specimens. As for the manufacturing, the testing of the IFBT specimens was realised in the two different laboratories. The respective protocols used are described below.

Protocol A (UFC)

For each condition, tensile tests were conducted on at least five IFBT specimens using a MTS Criterion 45 universal machine, with a crosshead displacement rate of 1 mm/min and a load cell of 100kN. The longitudinal strain was measured with an Instron 2620-601 extensometer with a gauge length of 50 mm.

Protocol B (KU Leuven)

Tensile tests were performed on at least five specimens using a Zwick/Roell Z100 universal testing machine equipped with a 100kN load cell and a displacement rate of 2 mm/min. The longitudinal strain was measured with optical and clip on extensometers with a gauge length from 50 to 80 mm.

The small difference in displacement rate (factor 2) is supposed to have neglectable effect on the measured properties. Indeed, it was previously demonstrated for unidirectional flax epoxy composite that the measured tensile properties are significantly different only when the displacement rate is changed by a factor of 10, in the considered displacement range [37].

2.3.3 Back-calculation of fibre properties from IFBT tests

To correctly implement and exploit the IFBT tests, the selected matrix should have a high ductility so that the failure strain of the matrix is higher than that of the fibres. The mechanical properties of the epoxy systems used are synthetized in Table 2.

Table 2: Mechanical properties of the epoxy systems used for the IFBT specimens

Epoxy systems	**E-modulus (GPa)**	**Stress at failure (MPa)**	**Strain at failure (%)**
GreenPoxy 56 / SD 7561	2.5	60	5
Epikote 828 LVEL + Dytek DCH-99	2.7	70	4

Protocol A (UFC)

The effective longitudinal modulus of the fibres (E_f) was obtained by back-calculation using the rule of mixtures proposed by Madsen et al. [38] for plant fibre composites (Eq. 4):

$$E_f = \frac{E_c - E_m . V_m}{\eta_0 . \eta_1 . V_f} \quad (4)$$

where E_C is the composite modulus, E_m the matrix modulus, η_0 the fibre orientation factor, η_1 the fibre length factor. In this work, the fibre length factor (η_1) was considered equal to 1 (which is generally the case when the length to diameter ratio of the fibres is higher than 50). η_0 is equal to 1 for all the specimens except for the roving which has a twist level of approximately 35 turns/meter. In this case, the fibre orientation factor is calculated using equation 5.

$$\eta_0 = \cos^2(2\alpha) \quad \text{with } \alpha = tan^{-1}(2\pi r T) \quad (5)$$

where α is the surface twist angle, r the radius of roving and T the twist level of roving.

Taking into account the non-linear tensile behaviour generally observed for plant fibre composites, the composite modulus was measured on two different strain ranges: Ec_1 between 0 and 0.1% of longitudinal strain and Ec_2 between 0.3 and 0.5%. The corresponding moduli determined by back-calculation at the scale of the fibres were noted E_{f1} and E_{f2}.

The effective longitudinal tensile strength of the fibres (σ_f) was obtained by back-calculation using the equation suggested in [39, 40] for plant fibre composites (Eq. 6):

$$\sigma_f = \frac{\sigma_c - \sigma_m . V_m}{\eta_0 . \eta_1 . \eta_d . V_f} \quad (6)$$

with σ_c the stress at failure of the composite, σ_m the stress in the matrix at the failure strain of the composite, η_d the fibre diameter distribution considered to be equal to 1 in this study.

Protocol B (KU Leuven)

The effective stiffness and strength of the fibres were determined as in Bensadoun et al. [16]. The equations used are given below. Again, stiffness was determined in two strain intervals.

$$E_f = \frac{E_c - E_m . (1 - V_f)}{V_f} \quad (7)$$

$$\sigma_f = \frac{\sigma_c - \sigma_m . (1 - V_f)}{V_f} \quad (8)$$

2.3.4 Statistical analysis

ANOVA (Analysis of Variance) tests were performed to evaluate if the means of the measured mechanical properties of the tested batches were significantly different from each other. Most of the time, tests were realised on two batches to better discriminate the influence of one of the tested features (i.e., variety, year, sowing time, harvesting time, stem portion, scutching parameter and process stage). The confidence interval was fixed at 95%. For each test, a probability Pr was calculated. The difference between means is considered to be significant when Pr is inferior to 0.05. When more than two batches were compared at once, a single-step multiple comparison was preferred. A Tukey's test was used to evaluate if the means are significantly different from each other. It applies simultaneously to the set of all pairwise comparisons. Letters (a, b and ab) are used to report the results of the pairwise comparisons.

2.3.5 SEM observations

The cross-sections of the IFBT specimens were observed using a Scanning Electron Microscope TESCAN Mira3 operating at 20 kV. The specimens were embedded in a PMMA resin and polished with silicate paper (until fineness 2400).

3 Results and discussion

3.1 Tensile behaviour of IFBT specimens – non-linearity and scattering

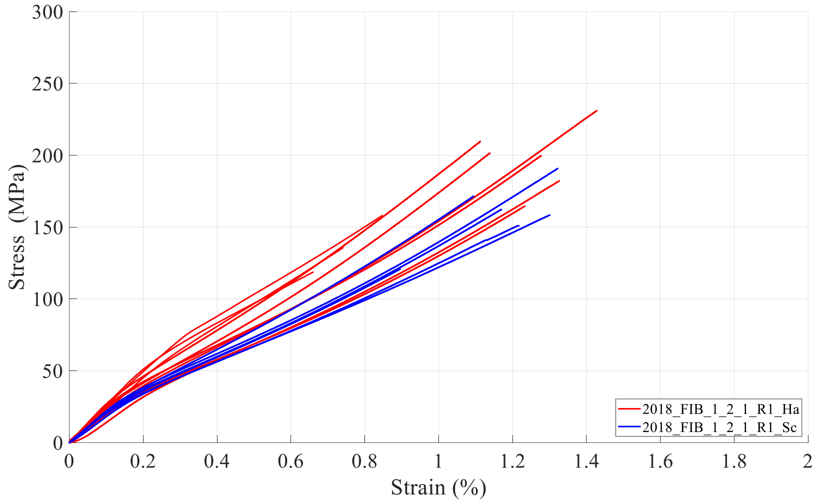

Figure 3: Tensile stress-strain curves of two tested batches of IFBT specimens pointing out the typical non-linear behaviour and scattering observed for the different tested batches

Figure 3 shows the typical tensile responses obtained for the IFBT specimens. Two tested batches are plotted. The shape of the tensile curves as well as the scattering of results within a same batch are representative of those observed for all the tested specimens. The tensile response is clearly non-linear with a linear response until a yield point located at a stress and strain level of approximately 40-50 MPa and 0.15-0.2%, respectively. This is typical of the unidirectional (UD) plant fibre composites and it was often documented for flax fibres [41-47]. However, a significant difference can be observed when compared to the typical bi-phasic behaviour reported for UD flax composites. Indeed, after the yield point a decrease in the apparent modulus is generally observed and this one remains almost constant up to failure. In the case of the tested UD hemp composites, a significant increase in the apparent modulus can be observed in the last stage of the tensile test. The difference of morphology (Figure 4), ultrastructure and the interface properties between hemp and flax fibres and the epoxy matrix can explain this difference in behaviour. This behaviour is similar to the observed at the scale of the individual hemp fibres [48, 49] often referred to as "type-3" behaviour. At the scale of individual fibres, the non-linear behaviour was attributed to complex phenomena involving stick-slip mechanisms and cellulose microfibrils reorientation in the fibre wall, and stress-induced crystallisation of the amorphous or pseudo-crystalline cellulose [49]. A non-linear tensile behaviour was also recently reported at the scale of the cellulose microfibrils themselves using molecular dynamics simulation [50]. Previous studies also pointed out that the shape of the fibre cross-section and in particular the degree of ellipticity has a strong effect on the shape of the nonlinearity of the tensile response [27]. This morphologic effect was demonstrated to be strongly related and coupled to structural parameters and physical mechanisms, such as the cellulose microfibrillar angle and the viscoelastic behaviour of the material of the fibre wall. The observed behaviour at the scale of the IFBT specimens could then result from the fibre behaviour and the difference observed when compared to flax could be attributed to the fibres' geometry. Figure 4 clearly shows the complex morphology of the hemp fibres, in particular when compared to flax.

Figure 3 also shows that the scattering within a same batch is quite limited in the first part of the curve and then increases with the increasing strain. This is attributed to the initial defects in the composite and the propagation of damage under tensile loading in the second part of the test. Indeed, initial cracks are often observed in IFBT specimens (Figure 4) due to the presence of impurities and remaining bark tissues.

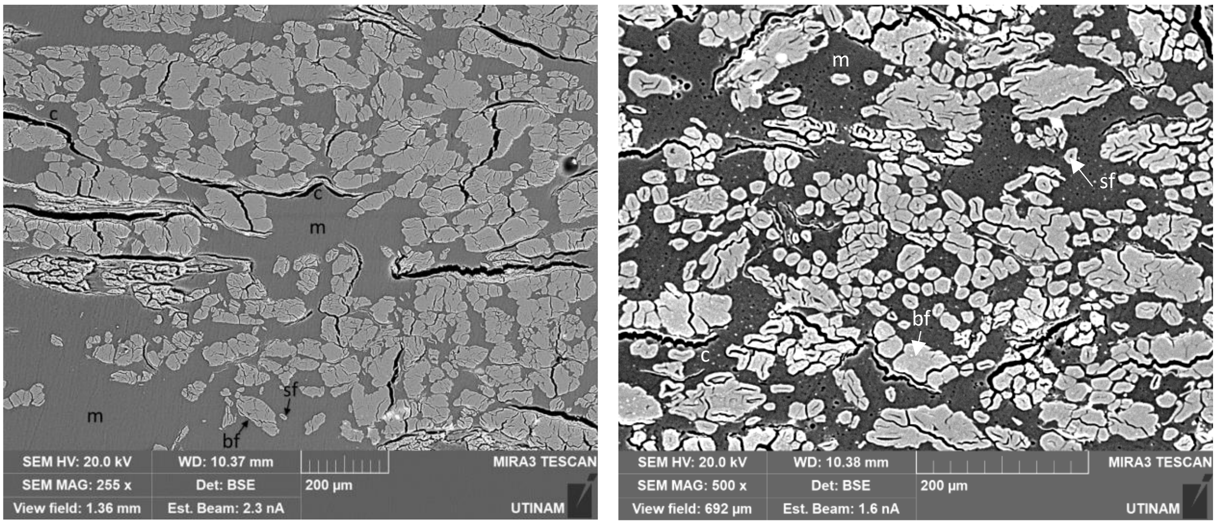

Figure 4: SEM image of the cross-section of an IFBT specimen showing the typical microstructure of the hemp composite (left) and SEM image of the cross-section of a specimen showing the typical microstructure of the flax composite (right). m: matrix, sf: single fibre, bf: bundle of fibres, c: cracks

The tensile properties measured at the scale of the IFBT specimens are synthetized in the table in supplementary information. The coefficients of variation of E_{c1}, E_{c2} and σ_c are in the range of 1-19%, 1-15% and 3-23%, respectively. This scattering in the tensile properties is attributed to the heterogeneities in the spatial distribution of fibres within and between samples, the porosity level, the presence of impurities in fibres (remaining shives and/or pieces of bark) and the geometrical defects of IFBT specimens.

Table 3: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of IFBT protocol (A and B). Letters (a, b and ab) are used to report the results of the pairwise comparisons.

LABORATORY	E_{f1}	E_{f2}	σ_f
2018_FIB_1_2_1_R1_Sc A	50.5 ^{a}	28.2 ^{b}	369 ^{a}
2018_FIB_1_2_1_R1_Sc B	46.1 ^{a}	23.4 ^{a}	449 ^{b}
Pr > F(Model)	**0.098**	**0.026**	**0.005**
Significant	**No**	**Yes**	**Yes**
2018_FIB_1_2_1_R1_Ha A	56.8 ^{a}	32.1 ^{b}	423 ^{a}
2018_FIB_1_2_1_R1_Ha B	51.8 ^{a}	23.2 ^{a}	490 ^{a}
Pr > F(Model)	**0.227**	**0.002**	**0.114**
Significant	**No**	**Yes**	**No**
2018_FUT_1_2_1_R1_Sc A	50 ^{a}	24.8 ^{a}	420 ^{a}
2018_FUT_1_2_1_R1_Sc B	45 ^{a}	21.5 ^{a}	433 ^{a}
Pr > F(Model)	**0.294**	**0.201**	**0.768**
Significant	**No**	**No**	**No**
2018_FUT_1_2_1_R1_Ha A	53 ^{b}	27.2 ^{b}	443 ^{a}
2018_FUT_1_2_1_R1_Ha B	45.3 ^{a}	21.7 ^{a}	412 ^{a}
Pr > F(Model)	**0.015**	**0.016**	**0.477**
Significant	**Yes**	**Yes**	**No**

The values obtained for the same fibre batches by the two different laboratories were also compared. Results of the statistical tests are presented in Table 3. The differences in the mean values are about 8 to 15% for E_{f1}, 13 to 28% for E_{f2} and 3 to 22% for σ_f. Most of the differences in the mean values are not significant from a statistical point of view except for E_{f2}. For this latter quantity, some differences may be related to the gauge length used for the strain measurement which is different for the two laboratories. The appearance of heterogeneities in the strain fields during the last stage of the tensile test due to the propagation of damage could then induce discrepancy in measurements in particular when the monitored specimen's length is different. A significant difference is also observed in the fibre strength of the first batch (2018_FIB_1_2_2_R1_Sc), and in the E_{f1} for the fourth batch (2018_FUT_1_2_1_R1_Ha). It is worth mentioning that the fibre individualisation was not fully achieved for this batch after scutching and that strong heterogeneities were observed between the fibre packages. The influence of the laboratory on the IFBT results was also underlined in [16]. However, the observed differences in this present study are lower and most of the time not significant from a statistical point of view. The quantification of the scattering and uncertainties in the measurements was a crucial step before investigating the influence of agronomic and processing parameters on the back-calculated fibre properties.

3.2 Influence of agronomic parameters on the effective properties of fibres

The influence of the agronomic parameters on the fibre properties was investigated. The results of the statistical analysis are presented in Table 4.

Interestingly, no significant difference (except for E_{f2} for Fibror 79 variety) in the properties of the hackled fibres (in the form of bundles of hackled fibres or slivers) was observed between the years 2018 and 2019, and this for the two tested varieties. The E_{f1} modulus is comprised between 52 and 61 GPa, the E_{f2} modulus between 27 and 36 GPa and the strength between 400 and 500 MPa, for the two years. It shows that the mechanical performance of the processed fibres can be reproduced from year to year. On the contrary, a significant difference in stiffness was observed between the years 2018 and 2019 at the scale of the mixed rovings. This can be attributed to the diversity of the very different batches mixed, particularly in year 2018. However, this result should not be transposed directly to the material which could be marketed later because the processors have a great know-how in the mixing of materials during the production of rovings to ensure good homogeneity and quality and good reproducibility over years.

No significant difference between the two varieties, Fibror 79 and Futura 75, was observed, as well in 2018 for the scutched and hackled fibres extracted from the first stem meter, and harvested at seed maturity. On the contrary, a significant difference was observed between the two varieties in 2019, at least after sliver forming; the Fibror 79 variety performing better. This could be due to the level of retting. Indeed, Hendrickx [51] pointed out for flax that E_{f2} is significantly influenced by the extent of retting.

As already observed by Musio et al. [13], the harvest at full flowering provides slightly stiffer fibres when compared to harvest at seed maturity. For all the tested batches except for the property E_{f2} of the batches "Ms", the stem portion does not influence the fibre properties results.

Table 4: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of variety, year, harvest time and stem portion. Letters (a, b and ab) are used to report the results of the pairwise comparisons.

YEAR	E_{f1}	E_{f2}	σ_f
2018_FUT_1_2_1_R1_Ha	53 ^{a}	27.2 ^{a}	443 ^{a}
2019_FUT_1_2_1_R1_Sl	52.2 ^{a}	29.2 ^{a}	397 ^{a}
Pr > F(Model)	**0.744**	**0.499**	**0.168**
Significant	**No**	**No**	**No**
2018_FIB_1_2_1_R1_Sl	61.4 ^{a}	33.5 ^{a}	421 ^{a}
2019_FIB_1_2_1_R1_Sl	57.8 ^{a}	36.3 ^{b}	508 ^{a}
Pr > F(Model)	**0.07**	**0.022**	**0.067**
Significant	**No**	**Yes**	**No**
2018_MIX_R_A	59.6 ^{b}	43.2 ^{b}	616 ^{a}
2019_MIX_R_B	50.6 ^{a}	32.7 ^{a}	615 ^{a}
Pr > F(Model)	**<0.0001**	**<0.0001**	**0.945**
Significant	**Yes**	**Yes**	**No**
VARIETY	E_{f1}	E_{f2}	σ_f
2018_FIB_1_2_1_R1_Sc	50.5 ^{a}	28.2 ^{a}	368.7 ^{a}

2018_FUT_1_2_1_R1_Sc	50 [a]	24.8 [a]	420 [a]
Pr > F(Model)	**0.834**	**0.204**	**0.175**
Significant	**No**	**No**	**No**
2018_FIB_1_2_1_R1_Ha	56.8 [a]	32.1 [a]	422.9 [a]
2018_FUT_1_2_1_R1_Ha	52.9 [a]	27.2 [a]	442.9 [a]
Pr > F(Model)	**0.302**	**0.081**	**0.606**
Significant	**No**	**No**	**No**
2019_FIB_1_2_1_R1_Sl	57.8 [a]	36.3 [a]	508 [a]
2019_FUT_1_2_1_R1_Sl	52.2 [b]	29.2 [b]	397 [b]
Pr > F(Model)	**0.037**	**0.028**	**0.022**
Significant	**Yes**	**Yes**	**Yes**
HARVEST TIME	**E_{f1}**	**E_{f2}**	**σ_f**
2018_FUT_1_2_1_R1_Sc	50.0 [a]	24.8 [a]	420 [a]
2018_FUT_1_1_1_R1_Sc	58.4 [a]	32.3 [b]	318 [a]
Pr > F(Model)	**0.06**	**0.036**	**0.07**
Significant	**No**	**Yes**	**No**
2018_FUT_1_2_1_R1_Ha	53.0 [a]	27.2 [a]	443 [a]
2018_FUT_1_1_1_R1_Ha	62.8 [b]	34.7 [b]	491 [a]
Pr > F(Model)	**0.04**	**0.006**	**0.202**
Significant	**Yes**	**Yes**	**No**
2019_FUT_1_2_1_R1_Sl	52.2 [a]	29.2 [a]	397 [a]
2019_FUT_2_1_1_R1_Sl	64.3 [b]	39.3 [b]	379 [a]
Pr > F(Model)	**0.001**	**0.013**	**0.695**
Significant	**Yes**	**Yes**	**No**
STEM PORTION	**E_{f1}**	**E_{f2}**	**σ_f**
2018_FUT_1_1_1_R1_Sc	58.4 [a]	32.3 [a]	318 [a]

2018_FUT_1_1_2_R1_Sc	55.7 ^{a}	28.7 ^{a}	337 ^{a}
Pr > F(Model)	**0.451**	**0.240**	**0.597**
Significant	**No**	**No**	**No**
2018_FUT_1_1_1_R1_Ha	62.8 ^{a}	34.7 ^{a}	491 ^{a}
2018_FUT_1_1_2_R1_Ha	60 ^{a}	32.7 ^{a}	513 ^{a}
Pr > F(Model)	**0.455**	**0.441**	**0.642**
Significant	**No**	**No**	**No**
2019_FIB_1_2_1_R1_Sl	57.8 ^{a}	36.3 ^{a}	508 ^{a}
2019_FIB_1_2_2_R1_Sl	55.2 ^{a}	36 ^{a}	504 ^{a}
Pr > F(Model)	**0.191**	**0.816**	**0.927**
Significant	**No**	**No**	**No**
2019_FIB_1_2_1_R1_Ms	56.2 ^{a}	33.7 ^{a}	506 ^{a}
2019_FIB_1_2_2_R1_Ms	52.9 ^{a}	31.5 ^{b}	515 ^{a}
Pr > F(Model)	**0.112**	**0.009**	**0.852**
Significant	**No**	**Yes**	**No**

3.3 Influence of processing parameters on the effective properties of fibres

Table 5: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of scutching speed. Letters (a, b and ab) are used to report the results of the pairwise comparisons.

SCUTCHING SPEED	E_{f1}	E_{f2}	σ_f
2019_FUT_1_2_1_R1_Sl	52.2 ^{a}	29.2 ^{b}	397 ^{a}
2019_FUT_1_2_1_R2_Sl	58.4 ^{a}	38.4 ^{a}	445 ^{a}
2019_FUT_1_2_1_R2_Ms	51.1 ^{a}	32.2 ^{ab}	510 ^{a}
Pr > F(Model)	**0.065**	**0.016**	**0.169**
Significant	**No**	**Yes**	**No**
2019_FIB_1_2_1_R1_Sl	57.8 ^{b}	36.3 ^{b}	508 ^{a}

2019_FIB_1_2_1_R2_Sl	64.1 [a]	39.9 [a]	420 [a]
Pr > F(Model)	**0.010**	**0.037**	**0.054**
Significant	**Yes**	**Yes**	**No**
2019_FIB_1_2_1_R1_Ms	56.2 [a]	33.7 [a]	506 [a]
2019_FIB_1_2_1_R2_Ms	49.1 [a]	30.8 [a]	492 [a]
Pr > F(Model)	**0.105**	**0.097**	**0.824**
Significant	**No**	**No**	**No**

In 2019, the impact of the scutching speed was also evaluated for the two varieties after sliver forming and doubling/stretching (Table 5). The reduction of the scutching speed led to a slight but significant increase of E_{f2} after sliver forming for Futura 75, while both E_{f1} and E_{f2} were increased for Fibror 79. This difference is not anymore significant after doubling and stretching (certainly due to the increase in the fibre fineness). In all cases, no significant effect is observed on the fibre strength.

The reduction of the scutching speed therefore does not really change the tensile properties of hemp fibres. However, reducing the stem progression speed as well as the turbine speed was shown to be particularly interesting regarding the long fibre yield. This reduces the generation of scutching tows and as a consequence maximise the long fibre yield as shown by Gregoire et al. [14] at the laboratory scale. It is therefore necessary to adjust the processing speed so that to minimise the generation of tows, but it is also important to keep the processing speed relatively high to keep the fibre production rate sufficiently high. A compromise between the long fibre yield and the production rate has to be determined.

3.4 Influence of processing stages on the effective properties of fibres

Finally, the impact of the processing steps, including scutching, hackling, sliver forming, doubling and stretching, was investigated. Results of the statistical analysis are synthetized in Table 6. For the Fibror 79 variety cultivated and harvested in 2018, the E_{f1} modulus increases from 50.4 GPa to 61 GPa and the strength from 369 MPa to 513 MPa from the scutched state to the doubled/stretched one.

For the mixed batch realised in 2018, a significant improvement except for E_{f1} was also observed with a general increase of the effective properties, from 55.9 GPa to 60 GPa for E_{f1}, 37.6 GPa to 42.7 GPa for E_{f2} and the strength increased from 439 MPa to 616 MPa from the sliver state to the doubled/stretched one. So, the effective properties are significantly increased during the processing in particular during hackling and stretching steps. This is attributed to the increase in cleanness and fineness of the fibres, while the mechanical properties of the fibres all along the process are not globally decreased drastically. The cleanness and fineness of the fibre induce a better adhesion between the fibres and the matrix, an increased adhesion surface, a better spatial distribution and less initial damage in the composite following manufacturing. Gregoire et al. [16] demonstrated that an equivalent number of kink-bands is globally present in hemp fibres extracted using a soft laboratory or the more aggressive scutching/hackling process. However, the size of the kink-bands is larger in the fibres extracted when submitted to more aggressive process parameters. If the kink-bands area is larger, it may be expected that the zones of weakness in the fibres are increased and this has as effect to reduce the tensile properties of the fibres. The fibres considered in the present paper were extracted using the same industrial equipment and process parameters as in [17], and it is expected that they contain similar number and similar surface area of defects. With such an amount of defects, the tensile properties of the elementary fibres determined in [17] are still globally high with tensile strength of 600 MPa and tensile modulus of 40 GPa and the mechanical potential of the fibres sufficient for load bearing composites. In a different study, Grégoire et al. [9] showed that the number of kink-bands increases as a function of the process severity. As a consequence, the number of kink-bands and the size of kink-bands after hackling is higher than after scutching. But as the technical fibres are more separated, the reinforcement properties of the fibres determined from back calculation of composite properties remain equivalent. Figure 5 shows a significant increase in fibre fineness during hackling with an average fibre width coming down from 61.9 to 47 μm for Futura 75 and from 67.4 to 48.2 μm for Fibror 79, and a less marked but still significant ($p= 2.10^{-5}$) decrease during stretching with a mean fibre width decreasing from 49.1 to 45.9 μm.

Table 6: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of processing steps: scutching, hackling, sliver forming, doubling and stretching. Letters (a, b and ab) are used to report the results of the pairwise comparisons.

PROCESSING STAGES	E_{f1}	E_{f2}	σ_f
2018_FIB_1_2_1_R1_Sc	50.4 ^{b}	28.2 ^{a}	369 ^{b}
2018_FIB_1_2_1_R1_Ha	56.8 ^{ab}	32.0 ^{a}	423 ^{ab}
2018_FIB_1_2_1_R1_Sl	61.4 ^{a}	33.5 ^{a}	421 ^{ab}
2018_FIB_1_2_1_R1_Ms	60.9 ^{a}	33.9 ^{a}	513 ^{a}
Pr > F(Model)	**0.002**	**0.065**	**0.023**
Significant	**Yes**	**No**	**Yes**
2018_MIX_Sl	55.9 ^{a}	37.6 ^{a}	439 ^{a}
2018_MIX_MS	59.7 ^{a}	34.9 ^{a}	486 ^{a}
2018_MIX_R	60 ^{a}	42.7 ^{b}	616 ^{b}
Pr > F(Model)	**0.06**	**0.004**	**0.009**
Significant	**No**	**Yes**	**Yes**

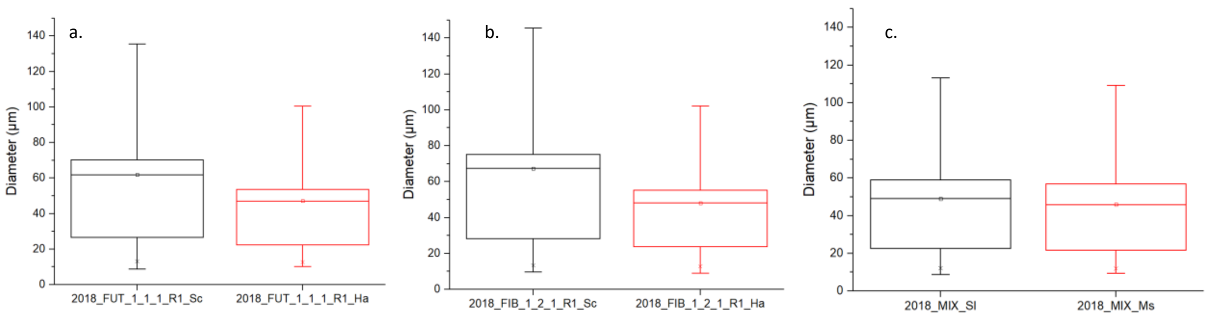

Figure 5: Influence of the processing stages on the fibre fineness. Influence of hackling evaluated on the Futura 75 (a.) and Fibror 79 (b.) varieties and influence of stretching characterised on the mixed batch formed in year 2018 (c.).

The improvement of the effective properties of long aligned fibres over the processing sequence is a major achievement since a detrimental effect of processing was observed in a previous work on long aligned hemp fibre [52]. Thygesen et al. [52] reported a monotonic decreasing relationship between the strength and the number of processing steps (including retting, scutching, carding and cottonisation). The fibre bundle strength was reported to be on average reduced by 27% per processing step at the applied conditions. It is also worth mentioning that the rovings type yarns obtained in the present study are suitable for weaving, without any traditional spinning step involving high twist of the fibres. The demonstration was made recently by Corbin et al. [53, 54] at the scale of woven balanced, unbalanced and quasi-unidirectional fabrics. Results also point out that the different stages of the hackling and scutching route up to the obtention of a low-twisted roving, as usually implemented for the production of yarns for textile applications can be simplified in view of composite applications.

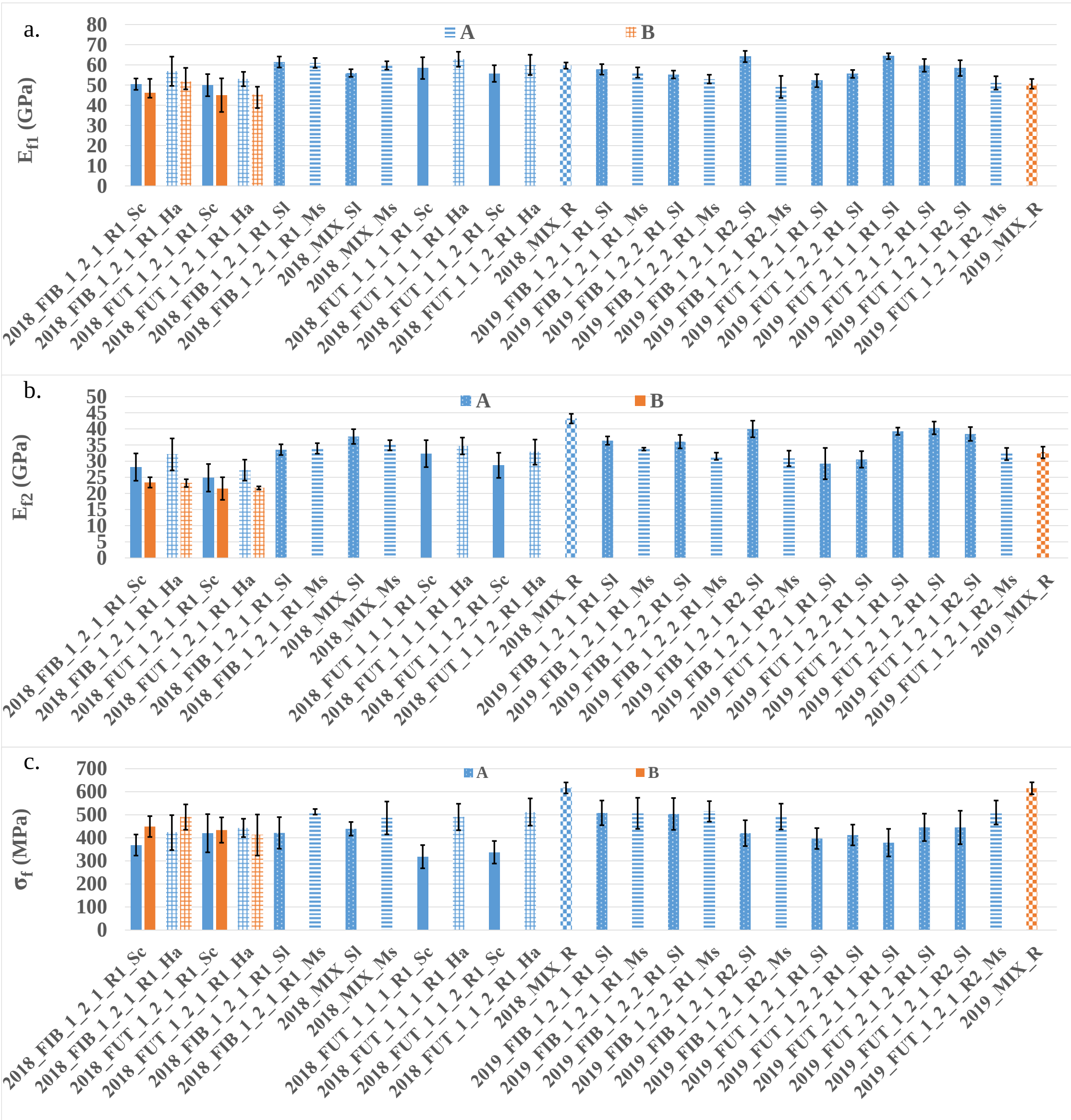

Figure 6: Tensile properties of the hemp fibres identified by inverse method from the IFBT tests, for the different tested conditions. a. E_{f1}, *b.* E_{f2} *and c. strength. The bar represents the mean value and the error bar the standard deviation; blue and orange represent the tests at the two participating laboratories.*

All the back-calculated fibre properties are finally synthetized in Figure 6. Results underline that whatever the agronomic and processing parameters considered, the effective fibre tensile stiffness (modulus Ef1) and strength are comprised between 45 and 64 GPa and 320 and 616 MPa, respectively.

These results can be compared to the ones published in literature for hackled flax and hemp fibres from previous studies (Figure 7). It clearly points out that hemp can achieve properties comparable to high quality long flax fibres for high performance composites, to be compared to on average 59.8 GPa and 527 MPa previously measured for industrially hackled flax in the frame of a round robin test [16].

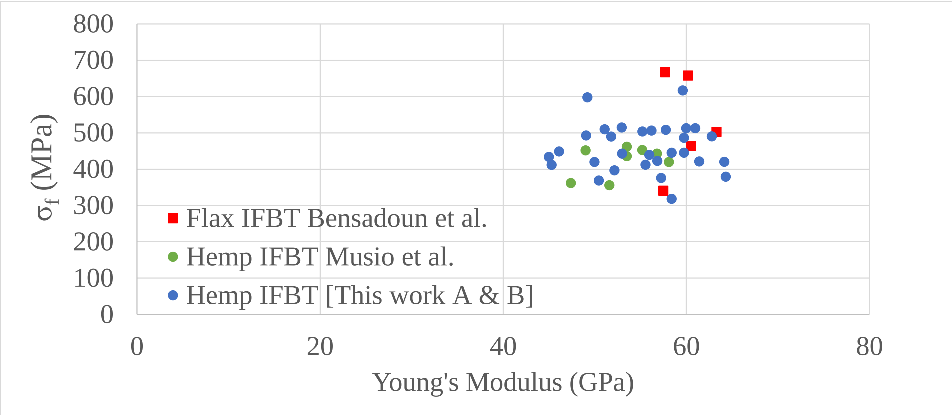

Figure 7: Effective tensile stiffness and strength of hemp fibres identified by inverse method from IFBT tests. Comparison to data from literature for hemp and flax fibres.

4 Conclusion

In this study, the influence of the processing stages and settings on the effective properties of fibres were investigated for the two cultivated hemp varieties, namely Futura 75 and Fibror 79, using textile flax machinery. Tests were realised on hemp straws cultivated, harvested and retted in Italy in 2018 and 2019. The sowing and harvest times as well as the stem portion were considered in the analysis. The mechanical properties of the long-aligned fibres obtained for the different batches at the different stages of the processing were evaluated using IFBT (impregnated fibre bundle test).

No significant difference in the effective properties of the fibres extracted in the first and second meter of the stems was observed. It means that the whole stem can be valorised for composite application. It is an important output in particular to maximise the fibre yield through processing and to ensure an economically viable cultivation and transformation of hemp straws.

Comparable properties were obtained for the two cultivation years demonstrating that a good fibre quality can be achieved from year to year.

As already observed in literature, the harvest at full flowering provides slightly stiffer fibres when compared to harvest at seed maturity. However, the effective properties obtained at seed maturity are still suitable for composite applications. This option allowing the seed harvesting would guarantee more income to the farmers and thus a more prosperous and profitable valorisation of the hemp production.

Interestingly, a good preservation of the effective fibre properties was observed over the processing steps and even an improvement was seen during hackling and stretching. This is attributed to both the conservation of the integrity of the fibres and the improvement of their individualization.

Overall, results point out that with a well-controlled retting and well-suited processing settings, hemp can achieve effective properties comparable to high quality long flax fibres. The fibre quality is suitable for the production of low-twisted rovings that can be further used for weaving. This work also suggests that the different stages of the scutching and hackling route up to the manufacturing of a low-twisted roving type yarn, as usually implemented for the production of yarns for textile applications could be shortened in view of composite applications as the final spinning step involving high twist of the fibres is not desired.

To validate the results presented in this work, future research should be carried out in different environmental conditions as these can affect fibre quality during plant growth and particularly during the phase of dew-retting.

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