

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

3 Xavier Gabrion¹, Gilles Koolen², Marie Grégoire³, Salvatore Musio⁴, Mahadev Bar³, Debora Botturi⁵,
4 Giorgio Rondi⁵, Emmanuel de Luycker³, Stefano Amaducci⁴, Pierre Ouagne³, Aart Van Vuure² and
5 Vincent Placet^{1*}

6 ¹ University of Bourgogne Franche-Comté, FEMTO-ST Institute, CNRS/UFC/ENSMM/UTBM,
7 Department of Applied Mechanics, F-25000 Besançon, France

8 ² Department of Materials Engineering, KU Leuven, B-3001, Heverlee, Belgium

9 ³ Laboratoire Génie de Production, LGP, Université de Toulouse, INP-ENIT, Tarbes, France

10 ⁴ Department of Sustainable Crop Production, Università Cattolica del Sacro Cuore, Piacenza, Italy

11 ⁵ Linificio e Canapificio Nazionale, Villa d'Almè, Italy

12 * Corresponding author

13 E-mail address: xavier.gabrion@femto-st.fr

14 ***Abstract***

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

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

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28

29 **1 Introduction**

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

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

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

42 Hemp is an environmentally friendly and fast-growing annual crop. It is thereby a substantial
43 consumer of carbon dioxide with an absorption of approximately 1.4 to 1.6 t of CO₂ per tonne of hemp
44 [3, 4]. With a yield average of 5.5 to 8 t ha⁻¹, this represents 9 to 13 tonnes of CO₂ absorption per
45 hectare harvested [5]. In that respect, hemp provides a carbon-negative material for engineering. Hemp
46 also requires limited amount of water to be produced [6]. Due to its vigorous growth, shading capacity
47 and disease resistance, hemp can be grown without the use of herbicide, pesticide or fungicide. It also
48 regenerates and improves the quality of soils. Inputs of fertilisers are low [7] and the interventions and
49 manpower requirements for farming are limited. The resulting energy cost for raw hemp fibre
50 production is estimated at approximately 5 GJ t⁻¹, about 7 times less than for glass fibres [3].

51 Expressed in CO₂ equivalents, the approximate production cost is only 680 kg eq. CO₂ t⁻¹ for hemp
52 fibres in comparison to the 2500 and 4000 kg eq.CO₂ t⁻¹ required for example for the production of
53 respectively PP and PET fibres [3]. Thus, it constitutes an interesting alternative to mineral and
54 synthetic fibres.

55 In Europe, hemp is currently processed using mechanical systems, based on beating (hammer mills)
56 and or fast rotating nailed rollers (referred to as decorticators), which provide fibres in the form of

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

85 but at the present time, it is not possible to change the design of industrial scutching plants as these
86 process hemp only for experiments or small-scale productions and textile flax the very vast majority of
87 the time.

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

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

110 In FBTT [28], a collection of several fibres are connected in parallel with both ends clamped to a support.
111 While quite easy to implement for synthetic fibres which are produced in the form of continuous

112 monofilaments, this test is very challenging for plant fibres due to their finite length and the resulting
113 discontinuities within rovings and yarns.

114 The IFBT is a well-established method for carbon and glass fibres, standardised for continuous and
115 staple-carbon fibre yarns (ISO 10618:2004). It was also adapted a few years ago for natural fibres and
116 validated by a round robin exercise on flax fibres [16]. In this method, an unidirectional composite is
117 manufactured and loaded in the fibre direction. The fibre stiffness and strength are then identified by
118 inverse method from the measured composite properties and a micromechanical model, generally the
119 rule of mixtures. A good impregnation quality with a negligible content of residual voids is required
120 [29]. If initially conceived and used for tensile loading, its use under compressive loading has also
121 been investigated more recently for flax [30].

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

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

140 130.000 ha of cultivations and still now most of the European varieties have been bred from Italian
141 genotypes. The two selected varieties are both bred in France by Hemp-It and are relatively similar for
142 their cycle, habitus and productivity and they are both well adapted to Italian conditions. Interestingly,
143 Fibror 79 is a « yellow » variety, which is easier to process than traditional ones [35].
144 Tests were realised on hemp straws harvested and retted in 2018 and 2019. The sowing and harvest
145 times as well as the stem portion are considered in the analysis. The mechanical properties of the long-
146 aligned fibres obtained for the different batches at the different stages of the processing are evaluated
147 using IFBT.

148 **2 Materials and methods**

149 **2.1 Materials and processing**

150 *2.1.1 Hemp stems: field trials, varieties and harvesting*

151 Hemp straws were obtained from large-scale field trials carried out in Piacenza, Italy (45° 3' 9.436" N
152 9° 41' 34.742" E) in 2018 and 2019 with two monoecious varieties, a green one Futura 75 (FUT) and a
153 yellow one Fibror 79 (FIB). They were sowed with a target plant density of 150 plants m⁻². The field
154 was fertilized with 60 kg of nitrogen per ha.

155 In 2018, the sowing was carried out on 25th April with a seed rate of 50 kg/ha. Stems were harvested at
156 two times: at the end of flowering on 11th August, and at seed maturity on 28th September as a dual
157 valorisation increases the income of the farmer (H2). Stems from H1 were dew-retted for 5 weeks (until
158 18th September), which was judged as appropriate for a good retting level according to the colour of the
159 stems.

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

163 For the harvest, a prototype machine was used. It enabled an efficient cut of the stems and the
164 formation of swaths of aligned stems. The harvested plants were then laid in the field for the dew-
165 retting. A second prototype was used to cut the plants in 1-meter stem portions and to turn the swath to
166 improve retting homogeneity. The bottom and middle part of the plant, cut in 1-meter portion, were

167 baled separately. The meteorological data (rainfall and temperature) are added in the supplementary
168 information.

169 2.1.2 Fibre processing



170

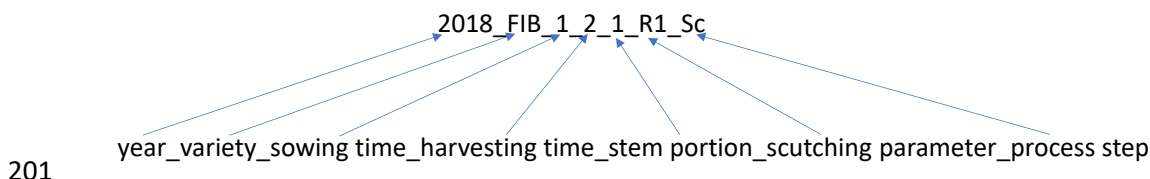
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Figure 1: Schematic representation of the processing stages and resulting products

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

181 At the end of the hackling line a continuous sliver with a large count (linear mass of about 15,000 tex)
182 was realised. This sliver was then processed into rovings at an industrial scale in Linificio e
183 Canapificio Nazionale (Villa d'Almè, Italy). The sliver was drawn and doubled several times and
184 slightly twisted to obtain at the end a roving of 350 Tex with a twist level of approximately 35
185 turn/meter. This process was also performed at the lab scale drawing system from Linimpianti
186 company (called Mini-Sytem), usually used in the industrial flax processing to evaluate the spinning
187 ability of scutched and hackled fibres. This was used in this study to evaluate if any difference in the
188 effective properties of the fibres can be detected between the matters processed with the Mini-System

189 (supposed to mimic the industrial process) and the industrial process itself. Indeed, the Mini-system
 190 simulates, at a reduced scale, the six drawing/doubling stages used in the flax spinning industry to
 191 prepare the slivers into rovings that will be used at the spinning stage. It consists of six drawing stages
 192 during which the linear mass of the sliver is decreased up to 150 Tex. During the different stages of
 193 this process, six parallel “Gill type” systems perform the different drawing operations. During this
 194 stage, the sliver mass is reduced but it is also homogenised as between each drawing stage, six drawn
 195 slivers are each time grouped together before the following drawing. During these operations, the
 196 technical fibre diameter may also be reduced when the technical fibres are pulled from the Gill system
 197 pins.
 198 The whole process is schematically represented in Figure 1. To facilitate the identification of the
 199 matter, the following label will be used (See Figure 2). The label fields and entries are described in
 200 Table 1.



202 *Figure 2. Identification of the matter.*

203 *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

204

205 The production of continuous reinforcement from the discontinuous hemp fibres requires a certain
206 minimum quantity. This quantity has not been systematically reached for all the batches. Therefore,
207 for the two tested years (2018 and 2019), some of the batches produced the same year were mixed
208 together to form a single sliver or roving. They were labelled 2018_MIX_SI, 2018_MIX_MS,
209 2018_MIX_R and 2019_MIX_R, respectively.

210 **2.2 IFBT specimens manufacturing**

211 IFBT specimens were prepared following the technical document “Impregnated Fibre Bundle Test –
212 IFBT – Methodology of uses” published by the European Confederation of Flax and Hemp (CELC)
213 [36]. This work being carried out within the framework of a European collaborative project, the
214 specimens were prepared in two different laboratories, labelled A and B, using the same above-
215 mentioned protocol and some small adjustments related to the know-how and previous experiences of
216 the involved research teams. The exact protocols are described below. For both, the IFBT specimens
217 were manufactured by aligning the long fibres obtained at the different stages of the processing and
218 impregnating them with an epoxy system.

219 **Protocol A (UFC)**

220 The specimens were polymerized from the GreenPoxly 56 resin and the SD 7561 hardener provided by
221 Sicomin company. The fibre samples were placed in the mould cavity after being conditioned at a
222 temperature of 23°C and a relative humidity of 50% during few hours to reach equilibrium. The resin
223 was poured on top of the fibres as the fibres were placed in the mould. The quantity of resin used was
224 calculated to reach a fibre volume fraction target in composites of approximately 40%. The counter-
225 mould was then placed on the top. No spacer was used to limit the porosity level. The specimens were

226 then cured at 60°C during 1h under a pressure of 2 bars and demoulded. A post-curing at 130°C was
227 then realised during 1h. The dimensions of the manufactured specimens were approximately 200 mm
228 x 16 mm x 1 mm. The manufactured IFBT specimens were then stored in a climatic chamber at 23°C
229 and 50% RH during a minimum of four weeks to reach the equilibrium moisture content. The
230 dimensions and mass of each specimen were measured. At least, six specimens were manufactured for
231 each tested condition.

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

$$234 \quad V_f = W_f \times \frac{\rho_c}{\rho_f} \quad (1)$$

$$235 \quad V_m = (1 - W_f) \times \frac{\rho_c}{\rho_m} \quad (2)$$

$$236 \quad V_v = 1 - V_f - V_m \quad (3)$$

237 where W_f is fibre weight fraction determined as the ratio of the measured mass of fibres to the
238 measured mass of composite, ρ_c , ρ_f and ρ_m specific gravity of composite, fibre and matrix,
239 respectively. ρ_c was determined as the ratio of the measured mass of composite to the measured
240 volume of composite and ρ_f and ρ_m were previously determined by pycnometry with values equal to
241 1.503 g/cm³ and 1.17g/cm³, respectively.

242 **Protocol B (KU Leuven)**

243 Protocol B is similar to A except for the following points. An Epikote 828 LVEL/ Dytex DCH-99
244 epoxy system was used. The fibres were dried during at least 24h at 60°C before the manufacturing of
245 the IFBT specimens. The specimens were cured at 150°C during 2h in a manual hydraulic press.

246 Spacers were used in the mould to approach a specimen thickness of 2mm. They were then
247 conditioned for at least one month at 21°C and 54% RH above a salt solution. The dimensions of the
248 manufactured specimens were approximately 200 mm x 10 mm with a thickness varying between 1.6
249 and 2 mm.

250 **2.3 Testing methods**

251 2.3.1 *Fibre fineness*

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

256 No particular pre-treatment (temperature or humidity stabilization, etc.) was carried out on the fibres.

257 The technical fibres were only cut to a fixed length before being fed into the scanner.

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

262 2.3.2 *IFBT*

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

266 **Protocol A (UFC)**

267 For each condition, tensile tests were conducted on at least five IFBT specimens using a MTS
268 Criterion 45 universal machine, with a crosshead displacement rate of 1 mm/min and a load cell of
269 100kN. The longitudinal strain was measured with an Instron 2620-601 extensometer with a gauge
270 length of 50 mm.

271 **Protocol B (KU Leuven)**

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

276 The small difference in displacement rate (factor 2) is supposed to have neglectable effect on the
277 measured properties. Indeed, it was previously demonstrated for unidirectional flax epoxy composite

278 that the measured tensile properties are significantly different only when the displacement rate is
 279 changed by a factor of 10, in the considered displacement range [37].

280 2.3.3 Back-calculation of fibre properties from IFBT tests

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

284 *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 LEVEL + Dytek DCH-99	2.7	70	4

285

286 **Protocol A (UFC)**

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

$$289 \quad E_f = \frac{E_c - E_m \cdot V_m}{\eta_0 \cdot \eta_1 \cdot V_f} \quad (4)$$

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

$$295 \quad \eta_0 = \cos^2(2\alpha) \quad \text{with } \alpha = \tan^{-1}(2\pi r T) \quad (5)$$

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

297 Taking into account the non-linear tensile behaviour generally observed for plant fibre
 298 composites, the composite modulus was measured on two different strain ranges: E_{c1} between 0 and
 299 0.1% of longitudinal strain and E_{c2} between 0.3 and 0.5%. The corresponding moduli determined by
 300 back-calculation at the scale of the fibres were noted E_{f1} and E_{f2} .

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

$$303 \quad \sigma_f = \frac{\sigma_c - \sigma_m \cdot V_m}{\eta_0 \cdot \eta_1 \cdot \eta_d \cdot V_f} \quad (6)$$

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

306

307 **Protocol B** (KU Leuven)

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

$$310 \quad E_f = \frac{E_c - E_m \cdot (1 - V_f)}{V_f} \quad (7)$$

$$311 \quad \sigma_f = \frac{\sigma_c - \sigma_m \cdot (1 - V_f)}{V_f} \quad (8)$$

312 2.3.4 *Statistical analysis*

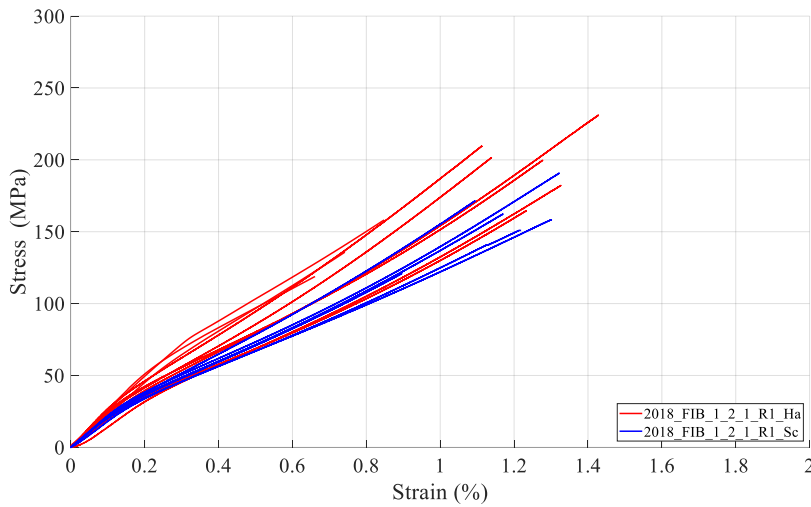
313 ANOVA (Analysis of Variance) tests were performed to evaluate if the means of the measured
314 mechanical properties of the tested batches were significantly different from each other. Most of the
315 time, tests were realised on two batches to better discriminate the influence of one of the tested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

323 2.3.5 *SEM observations*

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

327 **3 Results and discussion**

328 **3.1 Tensile behaviour of IFBT specimens – non-linearity and scattering**

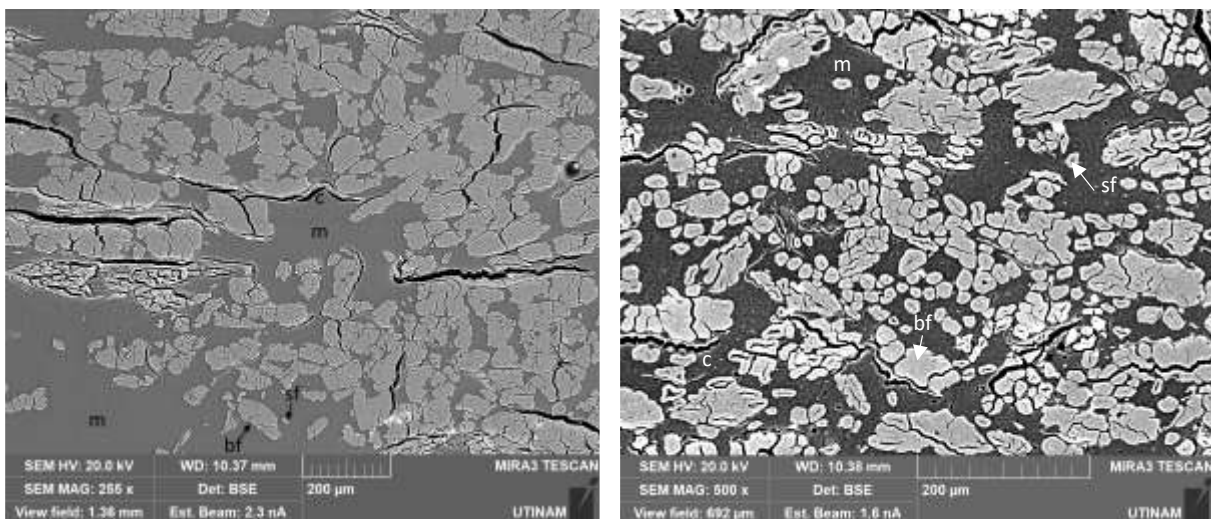


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330 *Figure 3: Tensile stress-strain curves of two tested batches of IFBT specimens pointing out the typical non-linear behaviour*
331 *and scattering observed for the different tested batches*

332 Figure 3 shows the typical tensile responses obtained for the IFBT specimens. Two tested batches are
333 plotted. The shape of the tensile curves as well as the scattering of results within a same batch are
334 representative of those observed for all the tested specimens. The tensile response is clearly non-linear
335 with a linear response until a yield point located at a stress and strain level of approximately 40-50
336 MPa and 0.15-0.2%, respectively. This is typical of the unidirectional (UD) plant fibre composites and
337 it was often documented for flax fibres [41-47]. However, a significant difference can be observed
338 when compared to the typical bi-phasic behaviour reported for UD flax composites. Indeed, after the
339 yield point a decrease in the apparent modulus is generally observed and this one remains almost
340 constant up to failure. In the case of the tested UD hemp composites, a significant increase in the
341 apparent modulus can be observed in the last stage of the tensile test. The difference of morphology
342 (Figure 4), ultrastructure and the interface properties between hemp and flax fibres and the epoxy
343 matrix can explain this difference in behaviour. This behaviour is similar to the observed at the scale
344 of the individual hemp fibres [48, 49] often referred to as “type-3” behaviour. At the scale of
345 individual fibres, the non-linear behaviour was attributed to complex phenomena involving stick-slip
346 mechanisms and cellulose microfibrils reorientation in the fibre wall, and stress-induced crystallisation
347 of the amorphous or pseudo-crystalline cellulose [49]. A non-linear tensile behaviour was also recently

348 reported at the scale of the cellulose microfibrils themselves using molecular dynamics simulation
 349 [50]. Previous studies also pointed out that the shape of the fibre cross-section and in particular the
 350 degree of ellipticity has a strong effect on the shape of the nonlinearity of the tensile response [27].
 351 This morphologic effect was demonstrated to be strongly related and coupled to structural parameters
 352 and physical mechanisms, such as the cellulose microfibrillar angle and the viscoelastic behaviour of
 353 the material of the fibre wall. The observed behaviour at the scale of the IFBT specimens could then
 354 result from the fibre behaviour and the difference observed when compared to flax could be attributed
 355 to the fibres' geometry. Figure 4 clearly shows the complex morphology of the hemp fibres, in
 356 particular when compared to flax.
 357 Figure 3 also shows that the scattering within a same batch is quite limited in the first part of the curve
 358 and then increases with the increasing strain. This is attributed to the initial defects in the composite
 359 and the propagation of damage under tensile loading in the second part of the test. Indeed, initial
 360 cracks are often observed in IFBT specimens (Figure 4) due to the presence of impurities and
 361 remaining bark tissues.



362
 363 *Figure 4: SEM image of the cross-section of an IFBT specimen showing the typical microstructure of the hemp composite*
 364 *(left) and SEM image of the cross-section of a specimen showing the typical microstructure of the flax composite (right). m:*
 365 *matrix, sf: single fibre, bf: bundle of fibres, c: cracks*

366 The tensile properties measured at the scale of the IFBT specimens are synthesized in the table in
 367 supplementary information. The coefficients of variation of E_{c1} , E_{c2} and σ_c are in the range of 1-19%,
 368 1-15% and 3-23%, respectively. This scattering in the tensile properties is attributed to the
 369 heterogeneities in the spatial distribution of fibres within and between samples, the porosity level, the

370 presence of impurities in fibres (remaining shives and/or pieces of bark) and the geometrical defects of
 371 IFBT specimens.

372 *Table 3: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of IFBT protocol*
 373 *(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

374

375 The values obtained for the same fibre batches by the two different laboratories were also compared.

376 Results of the statistical tests are presented in Table 3. The differences in the mean values are about 8

377 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

378 not significant from a statistical point of view except for E_{f2}. For this latter quantity, some differences

379 may be related to the gauge length used for the strain measurement which is different for the two
380 laboratories. The appearance of heterogeneities in the strain fields during the last stage of the tensile
381 test due to the propagation of damage could then induce discrepancy in measurements in particular
382 when the monitored specimen's length is different. A significant difference is also observed in the
383 fibre strength of the first batch (2018_FIB_1_2_2_R1_Sc), and in the E_{f1} for the fourth batch
384 (2018_FUT_1_2_1_R1_Ha). It is worth mentioning that the fibre individualisation was not fully
385 achieved for this batch after scutching and that strong heterogeneities were observed between the fibre
386 packages. The influence of the laboratory on the IFBT results was also underlined in [16]. However,
387 the observed differences in this present study are lower and most of the time not significant from a
388 statistical point of view. The quantification of the scattering and uncertainties in the measurements
389 was a crucial step before investigating the influence of agronomic and processing parameters on the
390 back-calculated fibre properties.

391 ***3.2 Influence of agronomic parameters on the effective properties of fibres***

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

394 Interestingly, no significant difference (except for E_{f2} for Fibror 79 variety) in the properties of the
395 hackled fibres (in the form of bundles of hackled fibres or slivers) was observed between the years
396 2018 and 2019, and this for the two tested varieties. The E_{f1} modulus is comprised between 52 and 61
397 GPa, the E_{f2} modulus between 27 and 36 GPa and the strength between 400 and 500 MPa, for the two
398 years. It shows that the mechanical performance of the processed fibres can be reproduced from year
399 to year. On the contrary, a significant difference in stiffness was observed between the years 2018 and
400 2019 at the scale of the mixed rovings. This can be attributed to the diversity of the very different
401 batches mixed, particularly in year 2018. However, this result should not be transposed directly to the
402 material which could be marketed later because the processors have a great know-how in the mixing
403 of materials during the production of rovings to ensure good homogeneity and quality and good
404 reproducibility over years.

405 No significant difference between the two varieties, Fibror 79 and Futura 75, was observed, as well in
 406 2018 for the scutched and hackled fibres extracted from the first stem meter, and harvested at seed
 407 maturity. On the contrary, a significant difference was observed between the two varieties in 2019, at
 408 least after sliver forming; the Fibror 79 variety performing better. This could be due to the level of
 409 retting. Indeed, Hendrickx [51] pointed out for flax that E_{f2} is significantly influenced by the extent of
 410 retting.

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

414 *Table 4: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of variety, year,*
 415 *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_SI	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_SI	61.4 ^a	33.5 ^a	421 ^a
2019_FIB_1_2_1_R1_SI	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_SI	57.8 ^a	36.3 ^a	508 ^a
2019_FUT_1_2_1_R1_SI	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_SI	52.2 ^a	29.2 ^a	397 ^a
2019_FUT_2_1_1_R1_SI	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

416

417 3.3 Influence of processing parameters on the effective properties of fibres

418 *Table 5: Results of the statistical analyses on the back-calculated fibre properties – Evaluation of the impact of scutching*
419 *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

420

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

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

434 **3.4 Influence of processing stages on the effective properties of fibres**

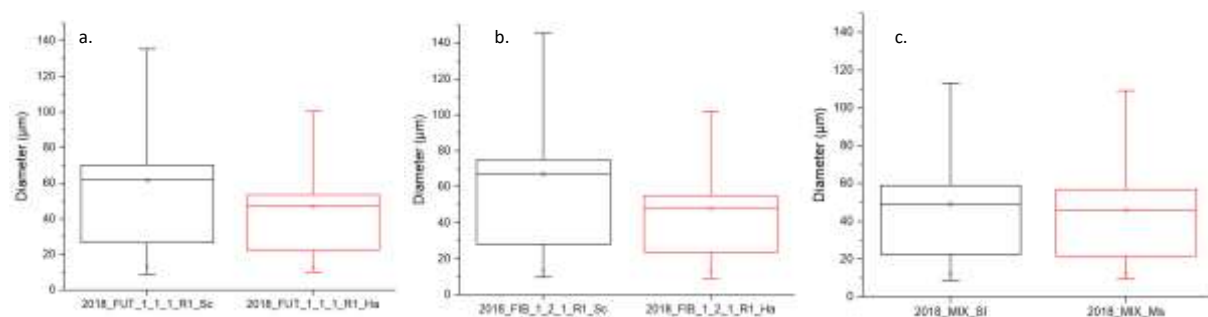
435 Finally, the impact of the processing steps, including scutching, hackling, sliver forming, doubling and
436 stretching, was investigated. Results of the statistical analysis are synthetized in Table 6. For the
437 Fibror 79 variety cultivated and harvested in 2018, the E_{f1} modulus increases from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

439 For the mixed batch realised in 2018, a significant improvement except for E_{f1} was also observed with
440 a general increase of the effective properties, from 55.9 GPa to 60 GPa for E_{f1} , 37.6 GPa to 42.7 GPa
441 for E_{f2} and the strength increased from 439 MPa to 616 MPa from the sliver state to the
442 doubled/stretched one. So, the effective properties are significantly increased during the processing in
443 particular during hackling and stretching steps. This is attributed to the increase in cleanness and
444 fineness of the fibres, while the mechanical properties of the fibres all along the process are not
445 globally decreased drastically. The cleanness and fineness of the fibre induce a better adhesion
446 between the fibres and the matrix, an increased adhesion surface, a better spatial distribution and less
447 initial damage in the composite following manufacturing. Gregoire et al. [16] demonstrated that an
448 equivalent number of kink-bands is globally present in hemp fibres extracted using a soft laboratory or
449 the more aggressive scutching/hackling process. However, the size of the kink-bands is larger in the
450 fibres extracted when submitted to more aggressive process parameters. If the kink-bands area is
451 larger, it may be expected that the zones of weakness in the fibres are increased and this has as effect
452 to reduce the tensile properties of the fibres. The fibres considered in the present paper were extracted
453 using the same industrial equipment and process parameters as in [17], and it is expected that they
454 contain similar number and similar surface area of defects. With such an amount of defects, the tensile
455 properties of the elementary fibres determined in [17] are still globally high with tensile strength of
456 600 MPa and tensile modulus of 40 GPa and the mechanical potential of the fibres sufficient for load
457 bearing composites. In a different study, Grégoire et al. [9] showed that the number of kink-bands
458 increases as a function of the process severity. As a consequence, the number of kink-bands and the
459 size of kink-bands after hackling is higher than after scutching. But as the technical fibres are more
460 separated, the reinforcement properties of the fibres determined from back calculation of composite
461 properties remain equivalent. Figure 5 shows a significant increase in fibre fineness during hackling
462 with an average fibre width coming down from 61.9 to 47 μm for Futura 75 and from 67.4 to 48.2 μm
463 for Fibror 79, and a less marked but still significant ($p= 2.10^{-5}$) decrease during stretching with a mean
464 fibre width decreasing from 49.1 to 45.9 μm .

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

468

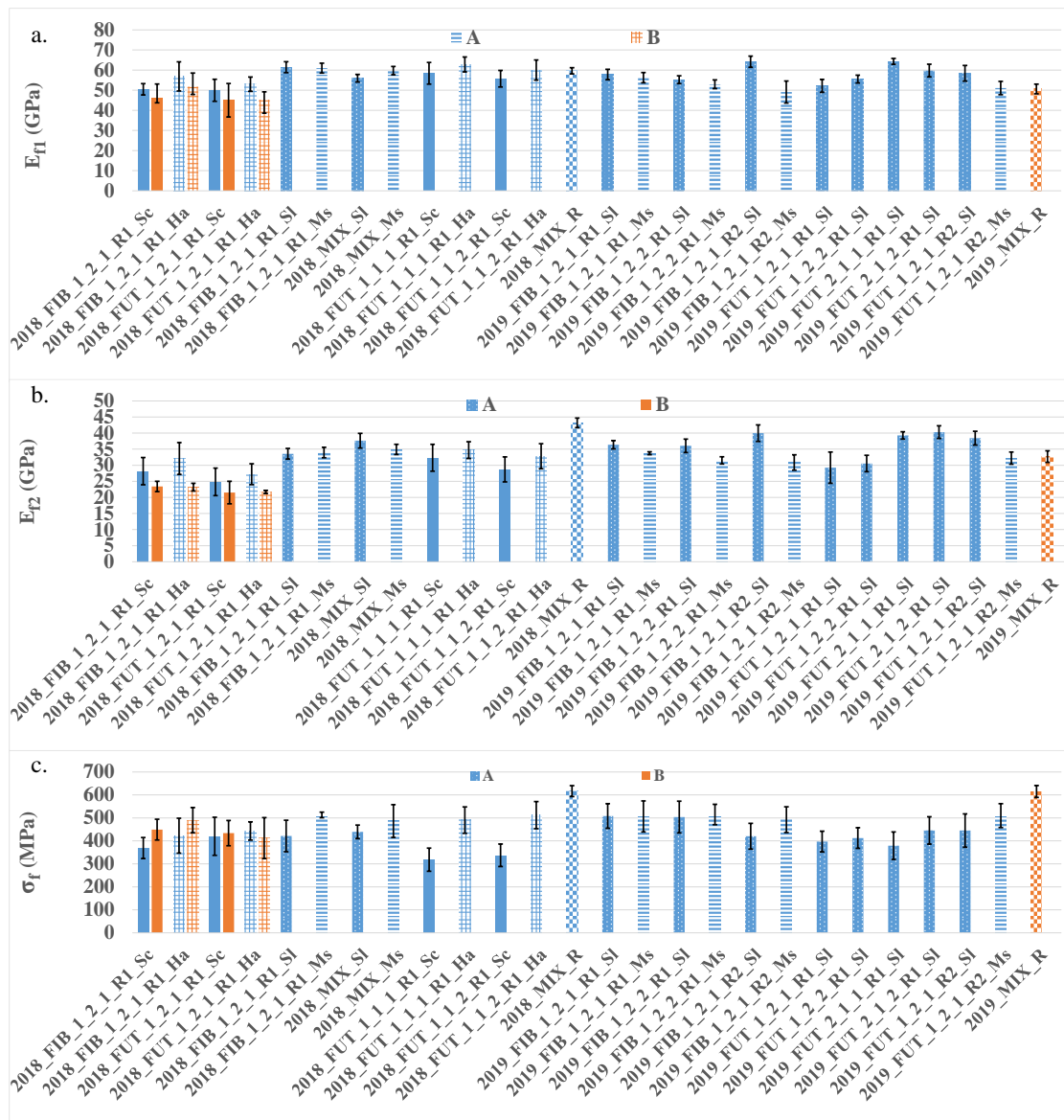


469

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

472 The improvement of the effective properties of long aligned fibres over the processing sequence is a
473 major achievement since a detrimental effect of processing was observed in a previous work on long
474 aligned hemp fibre [52]. Thygesen et al. [52] reported a monotonic decreasing relationship between
475 the strength and the number of processing steps (including retting, scutching, carding and
476 cottonisation). The fibre bundle strength was reported to be on average reduced by 27% per processing
477 step at the applied conditions. It is also worth mentioning that the rovings type yarns obtained in the
478 present study are suitable for weaving, without any traditional spinning step involving high twist of the

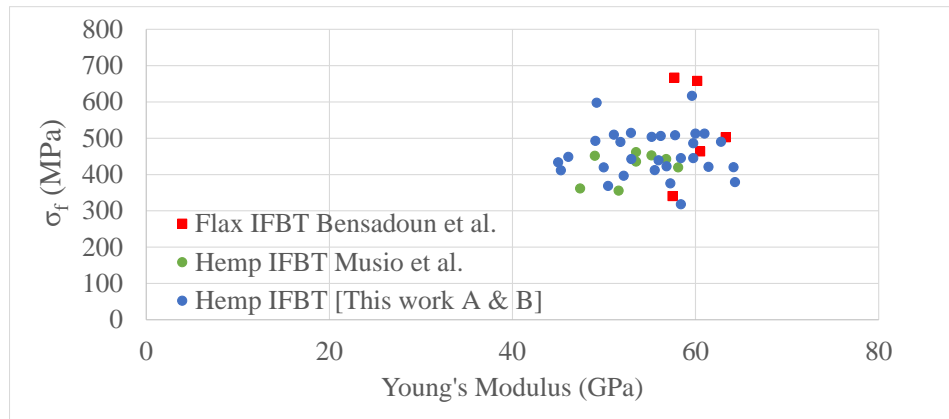
479 fibres. The demonstration was made recently by Corbin et al. [53, 54] at the scale of woven balanced,
 480 unbalanced and quasi-unidirectional fabrics. Results also point out that the different stages of the
 481 hackling and scutching route up to the obtention of a low-twisted roving, as usually implemented for
 482 the production of yarns for textile applications can be simplified in view of composite applications.



483
 484 *Figure 6: Tensile properties of the hemp fibres identified by inverse method from the IFBT tests, for the different tested*
 485 *conditions. a. E_{f1} . b. E_{f2} and c. strength. The bar represents the mean value and the error bar the standard deviation; blue*
 486 *and orange represent the tests at the two participating laboratories.*

487 All the back-calculated fibre properties are finally synthetized in Figure 6. Results underline that
 488 whatever the agronomic and processing parameters considered, the effective fibre tensile stiffness
 489 (modulus E_{f1}) and strength are comprised between 45 and 64 GPa and 320 and 616 MPa, respectively.

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



494
 495 *Figure 7: Effective tensile stiffness and strength of hemp fibres identified by inverse method from IFBT tests. Comparison to*
 496 *data from literature for hemp and flax fibres.*

497 **4 Conclusion**

498 In this study, the influence of the processing stages and settings on the effective properties of fibres
 499 were investigated for the two cultivated hemp varieties, namely Futura 75 and Fibror 79, using textile
 500 flax machinery. Tests were realised on hemp straws cultivated, harvested and retted in Italy in 2018
 501 and 2019. The sowing and harvest times as well as the stem portion were considered in the analysis.
 502 The mechanical properties of the long-aligned fibres obtained for the different batches at the different
 503 stages of the processing were evaluated using IFBT (impregnated fibre bundle test).
 504 No significant difference in the effective properties of the fibres extracted in the first and second meter
 505 of the stems was observed. It means that the whole stem can be valorised for composite application. It
 506 is an important output in particular to maximise the fibre yield through processing and to ensure an
 507 economically viable cultivation and transformation of hemp straws.
 508 Comparable properties were obtained for the two cultivation years demonstrating that a good fibre
 509 quality can be achieved from year to year.
 510 As already observed in literature, the harvest at full flowering provides slightly stiffer fibres when
 511 compared to harvest at seed maturity. However, the effective properties obtained at seed maturity are
 512 still suitable for composite applications. This option allowing the seed harvesting would guarantee

513 more income to the farmers and thus a more prosperous and profitable valorisation of the hemp
514 production.

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

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

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

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