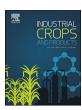
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Investigation of the potential of hemp fibre straws harvested using a combine machine for the production of technical load-bearing textiles



M. Grégoire^a, B. Barthod-Malat^{a,b}, L. Labonne^b, P. Evon^b, E. De Luycker^a, P. Ouagne^{a,*}

- ^a Laboratoire Génie de Production, LGP, Université de Toulouse, INP-ENIT, Tarbes, France
- ^b Laboratoire de Chimie Agro-industrielle (LCA), Université de Toulouse, INRA, INPT, Toulouse, France

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ABSTRACT

In western Europe, hemp is mainly cultivated for the seeds and the fibre rich straws, randomly aligned following the harvesting with a combine harvester. The straws are mainly valorised for low added value products such as pulp for paper and for insulation. They are also valorised in low property short fibre composites. With the view to use hemp fibres extracted from randomly aligned straws for higher added values, this work proposes to study a different process (fibre opener) for extracting the fibres than the one which is classically used in the industry (hammer mill) and to investigate the extraction performances and the impact of the process on the hemp morphological and mechanical properties.

The morphological and mechanical properties measured at different moments of the extraction indicate that, even in the less favourable case, the length of the fibres (~ 5 cm) is high enough for textile processing via the carded route. The tensile strength (~ 660 MPa) and modulus of elasticity of (38 GPa) of the individual hemp fibres are situated above most of the ones of fibres extracted mechanically using a hammer mill (630 MPa and 25 GPa for strength and modulus respectively) despite the fact that large quantities of kink band defects are observed on each fibre. This therefore shows that the "all fibre" opener is suitable for the production of fibres that can be considered for the manufacturing of technical textiles such as load-bearing woven geotextiles or midrange load-bearing composite reinforcements.

1. Introduction

Cannabis, the plant behind the hemp fibre is considered to find its origins in Central Asia, probably between the Caspian Sea and the south of Lake Baikal (De Candolle, 1884). Historic evidences show that the cannabis plant was cultivated in China since about 4000 years BC without interruption. The whole plant was valued. It was primarily grown for fibres and for textile as it constituted the main textile fibre resource in Northern China. The fibres were used for the production of textile fabrics, ropes, fish nets as well as for the production of paper. The seeds were also valued for the production of frying oil and the fruits, leaves and roots were used for traditional medicine and for hallucinogenic drug (Li, 1974). In Europe, Cannabis was probably introduced around 1500 BCE.

Traditional processing of hemp for fibres required large amounts of manual labour. It was considered in Asia or in Europe as a symbol of suffering. This is why farming and processing of hemp became more and more mechanized from the beginning of the 20th century. However, this was not sufficient to counter balance the emergence of chemical

fibres particularly after world war two (Clarke, 2010).

Large hemp cultivation and mechanized traditional processing remained in Eastern Europe. It is based on water retting and long fibre separation for the production of yarn. It uses hemp dedicated scutching and hackling equipments for the preparation of fibres. In Western Europe, the water retting is forbidden and only dew-retting can be performed. It is however a step that still needs to be optimised and globally only all-fibre processing is performed since low THC (the psycho-active substance) varieties were legalised in the European Union states between 1993 and 1996 (Carus, 2002). The different harvesting, fibre extraction and processing techniques were reviewed by Amaducci and Gusovius (2010).

As in ancient China, it is important that the whole hemp plant can be valued. Different ways of valorisation are described in the literature. It mainly concerns fibre and seed valorisation for oil or human consumption. Tang et al. (2016) investigated the possibility to grow hemp that would be suitable for seed and fibre valorisation. Other high value valorisation such as the extraction of pharmaceutical chemicals from hemp by-products such as leaves or bracts are also investigated

E-mail address: pierre.ouagne@enit.fr (P. Ouagne).

^{*} Corresponding author.

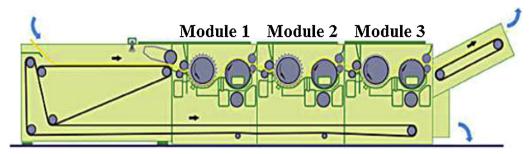


Fig. 1. Laroche Cadette 1000 fibre extraction device (from the Laroche Cadette company website).

(Calzolari et al., 2017). Other added value valorisation can also be encountered in the seed meal (Pojić et al., 2014).

In different reports (Carus and Sarmento, 2016, Carus, 2018), it was shown that the cultivation of hemp in Western Europe highly increased in the last years to reach a surface of about 33,000 ha in 2016 and 43,000 ha in 2017. In 2013, for 15,700 ha it represented a mass of 85,000 tons of straws that were separated in 25,000 tons of fibre, 43,000 tons of shives and 13,000 tons of vegetal dusts. 11,500 tons of seeds were also harvested as well as 240 tons of flowers and leaves for pharmaceutical, food and production of essential oil for food and beverages.

In Western Europe, seeds, straws and the rest of the plant need to be harvested. This is why, combine harvesting systems capable to harvest the seeds and cut the stems simultaneously were designed. Gusovius et al. (2016) reviewed the different devices that can be encountered either in experimental centres or on the market. Some experimental devices are capable to harvest the seeds and to mow and align the stems on the ground to favour dew-retting. Water or dew retting are favoured in Eastern Europe. The whole stems are then processed in dedicated hemp scutching and hackling facilities. In Western Europe, the hemp is generally harvested using modified cereal combine machine. In that case the seeds are harvested and the stems are cut and processed within the machine. The stems are often broken during this operation and therefore reduced in size. A windrow is formed at the back of the machine. The pieces of stem are randomly oriented within the windrow that can possess a thickness higher than 30 cm. This is not in favour of a homogeneous dew-retting. As dew-retting is generally not homogeneous or very low, aggressive techniques such as hammer mills are used in many cases to separate the vegetal fractions. The process is described by Xu et al. (2012). This process permits to separate the vegetal fractions of the stem (technical fibres, shives and vegetal dusts) but the fibres may be severely damaged. A lot of defects may be revealed at the surface of the fibres. These defects such as kink bands may be according to several authors at the origin of zones of weaknesses in the fibres (Baley, 2004; Bos et al., 2002; Symington et al., 2011; Yan

As hemp fibres were according to Carus and Sarmento (2016) mostly used for paper manufacturing (57 %), insulation (26 %) or for low resistance composite materials (14 %), maximising the mechanical properties of the fibres is not a first concern. As higher added value applications such as intermediate load-bearing composites or technical textiles such as geotextiles are targeted it is important that the mechanical properties of randomly oriented stems remain as high as possible. This work has therefore for goal to investigate the feasibility to separate the three vegetal fractions of the stems with an as low as possible impact on the fibre length and mechanical properties. A semiindustrial all-fibre opener is used with different process parameters and the performance of hemp fibres are measured and compared to the ones of fibres extracted manually so that to evaluate the impact of the process on the fibres. The mechanical properties of the hemp fibres are then related to the number of defects so that to investigate if a correlation exists between the number of defects observed at the different stages and a possible decrease in mechanical properties. A discussion

upon the possibility to use the extracted fibres for different technical applications is also be proposed.

2. Materials and methods

2.1. Plant material

The plant material used for the trials are obtained from hemp straws (*Cannabis Sativa*) of the Fedora 17 variety. The hemp stems are harvested with a combine machine at seed maturity. The hemp straws forming a windrow of randomly aligned pieces of stalks are left in the field for dew retting for a period of about three weeks. They are then pressed into round bales of about 300 kg for storage and transportation.

2.2. The fibre extraction device

A semi-industrial "all fibre" opening device (Laroche Cadette 1000) is used to separate the different vegetal fractions of the hemp plant stems. The fibres are extracted from the stems without re-wetting. The device is presented in the Fig. 1. It is located at the AGROMAT platform (Tarbes, France) which is the technological transfer hall of Laboratoire de Chimie Agro-industrielle (LCA).

The extraction device is used to open and clean the natural fibres and to obtain a lap of fibres, shives and vegetal dusts in three distinct compartments at the outlet. It consists of three modules. The raw material is fed into each module by a pair of rollers of which one is smooth and the other is grooved rubber. Each module has a cylinder equipped with nails, i.e. the fibre extraction roller, which has an adjustable rotation speed from 750 to 1800 rpm. The hemp shives fall by gravity during extraction, and they are evacuated by a conveyor belt. At the end of each module, a perforated suction roller is used to remove vegetal dust and small shives from the material, and also to transfer the forming lap to the next module or to the outlet. The vegetal dusts and the shives are sent to reception bags following their aspiration by the perforated rollers. Each perforated roller is equipped with a motor with a maximum rotation speed of 2865 rpm. As indicated in Table 1, this device is capable to process 175 kg of straw per h. Of course, this value depends on the size of the equipment. This one is a middle size equipment having a 1 m wide belt, used in our context to demonstrate its use to industrial companies that could opt for a larger equipment. The test parameters used during this study are presented in the Table 1.

2.3. Manual extraction of elementary fibres

To investigate the impact of the extraction process, it is important to determine the initial mechanical potential of the fibres. In this goal, fibres are extracted manually from the stems with the greatest care so that they are not damaged and represent as closely as possible their initial reinforcement potential. Hemp pieces of stems are extracted from the balls and the central part is then immersed in water at 30 $^{\circ}\text{C}$ for 72 h in order to facilitate the extraction of the elementary fibres. The elementary fibres are obtained by peeling the stems manually and by extracting them with the highest care from the peeled fibre assembly.

Table 1Test parameters used for the fibre extraction device.

| | Module 1 (M1) | Module 2 (M2) | | Module 3 (M3) |
|--|---------------------|---------------|---------------------|-----------------|
| Inlet flow rates (kg/h) | 175 | 175 | | 175 |
| Feed belt speed (m/min) | 3.5 (M1 inlet) | - | | |
| Transmission speed of the lap (m/min) | 2.2 (from M1 to M2) | | 1.5 (from M2 to M3) | |
| Speed of the conveyor belt (m/min) | _ | - | | 1.8 (M3 outlet) |
| Extracting roller rotation speed (rpm) | 725 | 725 | | 725 |
| Perforated roller rotation speed (rpm) | 1500 | 2000 | | 2000 |

2.4. Vegetal fractions

After processing the Laroche Cadette 1000 all fibre opening device, during 10 min, three vegetal fractions are obtained at the outlet of the extraction device (the lap, the shives and the vegetal dust collected by the perforated cylinders and on the belt). All the three vegetal fraction are weighed so that to determine the yields of the different constituents. However, the lap may still contain shives that are trapped in the fibres. Therefore, for each lap, the shives from the lap are separated manually from the fibres. The shives content inside the lap can then be determined and this permits to calculate the real fibre, shives and vegetal dust contents after extraction. The vegetal fractions obtained are shown in the Fig. 2. The analysis of the vegetal fractions is performed on three different batches at different stages of the extraction process: fibres extracted from the device after module 1 (M1), module 2 (M2) and module 3 (M3).

2.5. Morphological analysis

A morphological analysis of the technical fibres and shives obtained at the outlet of each module (M1, M2 and M3) of the fibre extraction device is carried out. First, from the lap, 100 fibres bundles are removed and their lengths are determined. Each bundle is attached to one end and then extended so as to know its actual length, which is measured between its two extremities.

The average diameters of the same fibre bundles are also determined. It is an average value calculated from 5 measurements determined along the bundle using an optical microscope (Olympus PMG3-F3. France).

Each beam is then weighed to calculate its single fibre linear density. The weighing of each of the bundle is carried out on a scale accurate to the thousands of g.

The shives obtained during the extraction of the fibres by the CADETTE device are morphologically analysed. A bulk sample of over 3000 hemp shives is scanned using an office device and analysed on the ImageJ image processing software so that a granulometric study of the shive size (length and width) is carried out.

2.6. Number of defects at the surface of the fibres

The defects observed at the surface of the fibres are mainly kink bands. These defects may be observed under polarised light as described by Baley (2004) for example. The number of defects is

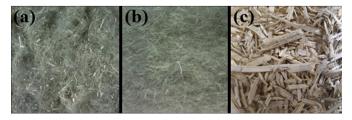


Fig. 2. Fibre lap (a), fibres after manual shive extraction (b), and shives obtained after the extraction (c).

determined on fibres glued on a paper frame, and subsequently used for tensile tests. The number of kink bands for each tested fibre is counted using an optical microscope under polarized light over a length of 330 $\mu m.$

2.7. Statistical analysis

In order to check if there is a significant difference in terms of average values, taking into account the standard deviation, a statistical test, named the two sample t-test, is performed. A confidence interval of 95 % is considered. In the columns of the tables, values with different superscript letters are considered statistically different.

2.8. Influence of the extraction steps on the physical and mechanical properties

An additional trial is performed on the batch. The extraction is carried out on the fibre extraction device for 10 min. Lap is collected after each module (module 1 M1, module 2 M2 and module 3, M3) and thirty elementary fibres are manually extracted from fibre bundles for each batch without pre-treatment. The morphological analysis of the bundle and the mechanical properties of the elementary fibres are determined following the recommendations of the standard test method NF T25-501-2 (AFNOR, 2015) in a large extent so that to investigate their evolution throughout the different fibre extraction modules and then to compare them with the initial properties of the elementary fibres at the field outlet. Similarly, the evolution of the number of defects (kink bands) according to modules M1, M2, M3 is analysed.

2.9. Determination of the cross-sectional areas of the elementary fibres

Thirty elementary fibres are extracted from each batch (raw material, module 1, module 2 and module 3). Each individual fibre is then positioned at each end in plastic tabs with a gauge length of 12 mm and glued into them with a photo-curing adhesive (DYMAX, Wiesbaden, Germany) to prevent slipping during morphological measurements and traction

The measurement of the cross-section areas of the elementary fibres is carried out by using an automated laser scanning method provided by the Dia-Stron company. A Fibre Dimensional Analysis System (FDAS) controlled by UV Win software (Diastron Ltd., Hampshire, UK) is used to determine individual fibre diameters using a high-precision laser photodetector (LSM 500S, Mitutoyo, Japan). The individual fibre mounted in the plastic tabs is positioned in jaws and held in position by a pneumatic system as shown in the Fig. 3. The fibre is scanned over its entire circumference at 360° as well as along its length. For this study, fibres with a gauge length of 12 mm are scanned ten times in different positions over their entire length. When rotating the fibre, the maximum and minimum diameters after each revolution are measured and mean values over the 10 measurements of the minimum and maximum diameters are computed. The other measurements correspond to projected diameters. The area of the fibre is then calculated from the maximum and minimum diameters by following an elliptical model. As Garat et al. explained in their article (Garat et al., 2018), such a model

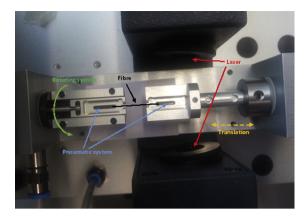


Fig. 3. Fibre dimensional analysis system.

gives the possibility to evaluate an area close to the real area of the technical or single fibres that are not necessarily cylindrical as it is assumed in the standard. This measuring system allows diameters to be determined with an accuracy of 0.01 μ m. However, it should be noted that this system does not measure concave surfaces. This due to the fact that Ombroscopy (Brillaud et al., 1996) is used to evaluate the fibre diameters and this technique only measures the projected shadow of the fibre. As a consequence, the projected shadow does only take into account the largest envelop of the fibre. The inner concavity cannot be analysed using this technique. This is however not a problem since the individual hemp fibres are globally convex [Bourmaud et al., 2018].

2.10. Tensile testing on elementary hemp fibres

Tensile tests are performed on thirty elementary fibres for each batch (manually extracted, M1, M2, M3). The same specimens used to count the number of defects are used to determine the mechanical properties of the single hemp fibres. The specimens, after the measurement of their cross-sections all over their length are mechanically tested using a specifically developed device for long natural fibres such as hair, from the Diastron company. The system used is an automated Lex high-precision extensometer (Lex 820, Diastron Ltd., Hampshire, UK) composed of 2 elements: a load cell with a capacity of \pm 20 N as well as a stepping motor for traction, shown in Fig. 4.

It is used for failing at low strain values. The displacement is obtained with an accuracy of 1 μ m. The individual fibres are tensile tested at a speed of 1 mm/min. The measuring points are recorded with a

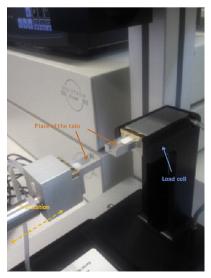


Fig. 4. Fibre tensile test system.

periodicity of 20 ms. This allows to calculate the tensile strength and the modulus of elasticity without using a supplementary strain measurement device.

2.11. Moisture content and chemical composition of the fibres, shives and vegetal dusts

Moisture contents are determined according to the standard ISO 665:2000 (ISO, 2000). Mineral contents are determined according to the standard ISO 749:1977 (ISO, 1997). The three parietal constituents, i.e. cellulose, hemicelluloses and lignins, are estimated thanks to the ADF-NDF (ADF stands for Acid Detergent Fibre, and NDF stands for Neutral Detergent Fibre) method of Van Soest and Wine (1967, 1968). Lastly, water-soluble compounds are determined by measuring the mass loss of the test sample after 1 h in boiling water. All the determinations are carried out in duplicates.

3. Results and discussion

3.1. Analysis of the vegetal fractions

As explained in paragraph 2.4, several plant fractions (lap, shives and vegetal dust) are obtained during the extraction with the Laroche Cadette 1000 fibre extraction device and their respective amounts are given in Table 2. The studies are carried out on a quantity of incoming material corresponding to 100 kg of dry hemp stems. The plant fractions (in mass) obtained from the Laroche Cadette after passing through the three extraction modules are 43 % of fibre lap, 49 % of shives and 8 % of vegetable dust.

However, the lap obtained from the device does not only contain fibres but also shives and a small amount of vegetal dust. The results given in the previous paragraph are therefore slightly biased because of this non-separation. In order to know the exact mass quantities of shives and fibres contained in the lap, mechanical sieving followed by manual sorting is carried out. The lap therefore contains 65 % of fibres and 35 % of other components (shives and vegetal dust).

These results indicate that it is important to have a supplementary stage at the extraction outlet in order to remove the remaining shives and thus obtain fibres sufficiently clean for technical textile application for example.

The actual quantities of the different plant fractions are obtained at the outlet of the fibre extraction device after sieving and manual sorting of the lap. The quantities of fibre extracted from the stems are 29~% while the quantities of shives are 57~%. The dust content is 14~%. By comparing the results obtained in this study to the ones presented in the study carried out by the French National Environment Agency (ADEME) (ADEME, 2011) it can be noted that they are very much comparable (29-32% of fibres).

It is interesting to note that the fibre amounts extracted with the same technique from randomly aligned linseed flax stems is superior to the one of hemp from this study as about 37 % of fibre, 54 % of shives and 9 % of vegetal dust were extracted (Ouagne et al., 2017). In Ouagne et al. (2017), it is mentioned that the proportion of fibres extracted from oleaginous flax straw was comparable to the one from textile flax (i.e. 40 % of the stem dry mass), and the latter is known for producing generally more fibres than hemp (30 % in mass) [ADEME, 2011]. In

Table 2Vegetal fractions collected at the outlet of each module.

| Batch | Total fibre content (%) | Total shives content (%) | Total dust content (%) |
|----------|-------------------------|--------------------------|------------------------|
| Module 1 | 59 | 36 | 5 |
| Module 2 | 59 | 31 | 10 |
| Module 3 | 65 | 20 | 15 |

addition, because shives from linseed and textile flax are less thick and therefore more brittle than those of hemp, it is reasonable to assume that part of this fraction is removed from the stems when the latter are packed into balls on the field, contributing to an artificial increase of the fibre proportion inside the treated batch of oleaginous flax straw used in that study. Lastly, Ouagne et al. (2017) rewetted the straw before the extraction of fibres, thus preserving the length of the technical fibres extracted (decrease in the fibre rigidity when re-wetted). This leads to fewer breakages along fibres, thus limiting the generation of fines (i.e. vegetal dust) and, consequently, increasing the fibre yield.

3.2. Influence of the extraction steps

As explained in paragraph 2.8, one of the goal of this study is to investigate the influence of the successive extraction modules of the Laroche Cadette 1000 fibre extraction device on several parameters such as the morphology of the bundles or the mechanical performance of the individual fibres.

3.2.1. Vegetal fractions collected at the outlet of each extraction module

The vegetal fractions obtained at the outlet of each module, M1, M2, M3 are studied. Pieces of lap are taken after each of the three modules. Sieving and final manual shive separation was performed. These three laps are weighed manually in order to determine their vegetal fractions: the fibre yields, the quantities of shives and vegetal dust that are trapped in the lap. The results obtained are presented in Table 6.

Table 6 shows the mass percentages of the different vegetal fractions extracted from the hemp laps after each module. The study shows that the fibre yield increases gradually over the three modules from 59 % to 65 % at the outlet of the fibre extraction device. The quantity of vegetal dust collected also increases from 5 % in module 1–15% in module 3 while, on the contrary, the shives rate decreases. With regard to the results of modules 1 and 2, it could be concluded that the decrease in the shives rate comes from their transformation into vegetal dust. Indeed, for a constant fibre content, the shives rate decreases while the dust rate increases. It is interesting to note that the passage into a second module of the machine does not lead to an increase in fibre yield. This is probably due to the fact that despite a reduction of the size of shives contributing to the observed generation of fines (vegetal dusts), the remaining shives are still large enough for being trapped in the fibre lap. The fibre purity of the lap is therefore not improved significantly.

On the contrary, from modules 2–3, an additional reduction in the size of hemp shives surely occurred in contact with the extraction roller in module 3 where the density of nails is much more important than in module 2 (2.3 nails per $\rm cm^2$ in module 3 instead of only 1.6 nails per $\rm cm^2$ in module 2). This probably allows the smaller particles of shives to fall down more easily by gravity from the fibre lap, being then evacuated by the dedicated conveyor belt situated at the bottom of the extraction device. This results in a better purity in technical fibres for the lap at the outlet of module 3 (from 59 % at the outlet of module 2–65%), simultaneously with a large decrease in its shives content (from 31 % to 20 %)."

3.2.2. Chemical analysis

The Laroche Cadette 1000 "All Fibre" extraction equipment generates three different fractions: technical fibers, shives (or chaff) and vegetal dust. The chemical composition of these three fractions is mentioned in Table 3.

The technical fibres are predominantly cellulosic (79.7 % of the dry matter), and this is expected. In parallel, because the chaff fraction constitutes the ligneous part of the hemp stems, it logically reveals a lower amount of cellulose (45.4 %) and, conversely, a significant content of lignins (21.2 %). Such lignin content is in perfect accordance with other studies in the literature reporting lignin contents inside hemp shives ranging from 20.7–24.5 % (Bag et al., 2011; Beaugrand

Table 3Moisture content (%) and chemical composition of fibres, shives and vegetal dusts (% of the dry matter).

| Batch | | Fibres | Shives | Vegetal dusts |
|------------------------------------|---|--|--|---|
| Miner Cellul Hemio Lignin | ure (%) als (% of the dry matter) ose (% of the dry matter) celluloses (% of the dry matter) as (% of the dry matter) -solubles (% of the dry matter) | 6.2 ± 0.3 1.1 ± 0.0 79.7 ± 0.9 6.0 ± 0.3 3.4 ± 0.9 9.2 ± 0.3 | 6.8 ± 0.2 1.0 ± 0.1 45.4 ± 0.2 26.2 ± 0.4 21.2 ± 0.2 5.4 ± 0.1 | 6.7 ± 0.1 4.1 ± 0.1 45.8 ± 0.3 19.8 ± 1.1 12.5 ± 0.8 10.0 ± 0.1 |
| | | | | |

et al., 2014; Brazdausks et al., 2016, 2017; Gandolfi et al., 2013) In the same way, the cellulose content of the hemp shives here obtained after the continuous extraction of technical fibres is also in agreement with other values reported in the literature (47 %) (Brazdausks et al., 2016, 2017). Looking at the vegetal dust, they are scarcely richer in cellulose (45.8 %) than shives, and they reveal median lignin content (12.5 %) between technical fibres (3.4 %) and shives (21.2 %). Thus, it is reasonable to assume that the vegetal dust originates first and foremost from shives. However, it is not impossible that some fines could also come from the localized breakdown of technical fibres, but to a lesser extent when looking at the cellulose content inside vegetal dust which remains quite low.

These chemical analyses confirm the hypothesis set out in paragraph 3.2.1. Indeed, the decrease in the shive rate seems to come primarily from their transformation into vegetal dust.

3.2.3. Morphological analysis

Morphological analyses are performed on 100 random bundles from each batch (module 1, module 2 and module 3). The average lengths of the bundles obtained are presented in Table 48. The results presented in Table 4 show a decrease in the average bundle length after passing through the different modules. It decreases from a length of 5.7 cm at the first module to a length of 4.7 cm and 3.8 cm at the second and third module respectively. The technical fibres are therefore partially broken as they pass through the modules of the fibre extraction device Laroche Cadette 1000. They can therefore turn into vegetal dust but to a low extent as the cellulose content measured in the vegetal dusts is very close to the one of the shives.

It can be observed on Fig. 5 that after the passage in module 1 and module 2 of the Laroche Cadette 1000 fibre extraction device there is not any bundle longer than 13 cm. The majority of the fibres are short in length with a high population for bundle lengths between 2 and 4 cm, whereas for bundles from other modules the majority of the lengths are between 1 and 5 cm. The lengths of hemp bundles obtained are generally equivalent to those of linseed flax (from 4 to 5 cm) (Ouagne et al., 2017). The standard deviations observed in Table 4 are high because a relatively large distribution of fibre length is observed. This is firstly due to the fact that short fibres are already present in the hemp stems. These are the tow fibres. Some of these technical fibres can exhibit values lower than 5 cm. The technical fibre length can also be reduced during the extraction process. As the all-fibre process used in this work is relatively aggressive, the long technical fibres are very much reduced. However, some of them remain long and this is why a large fibre distribution is observed with associated large standard dis-

The bundle length is globally sufficient for processing the fibres into

Table 4 Average fibre bundle lengths.

| Batch | Average fibre bundle length (cm) |
|----------|----------------------------------|
| Module 1 | 5.7 ± 4.4 |
| Module 2 | 4.7 ± 4.1 |
| Module 3 | 3.8 ± 2.2 |
| | |

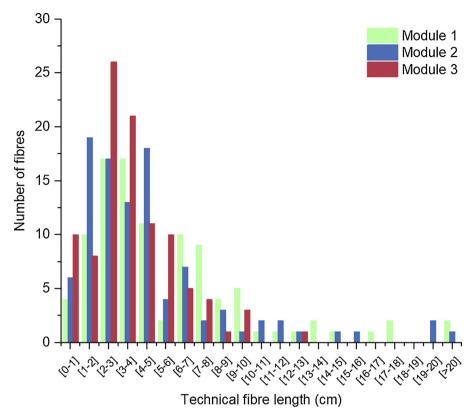


Fig. 5. Mean length of technical fibres after each extraction module.

technical yarns for mid-range load-bearing composite materials or for geo-textiles. In addition, when using a Laroche Cadette 1000 device, as in this study, it may not be necessary to use the three extraction modules if one wants to maintain the fibre length to the highest possible value.

3.2.4. Shives granulometry

As explained in paragraph 2.5, a morphological analysis is carried out on samples of approximately 3000 shives from the manual sorting carried out on the lap samples from modules 1, 2 and 3. After image processing on the ImageJ software, the shives lengths and widths are determined and their shape factor calculated. Table 5 presents the results obtained. The shape factor of the shives gradually decreases as they pass through the modules of the Laroche Cadette 1000 fibre extraction device, from 4.16 for module 1–3.25 for module 3. Fig. 6, which presents the shives distribution according to their lengths, shows that the majority of shives regardless of the module studied, are less than 5 mm long. Moreover, the graph shows that, except for the length range from 5 to 10 mm, the shives length decreases after passing through each extraction modules. This confirms once again the hypothesis of the transformation of shives into vegetal dust.

After module 3, some shives are still present in the lap as indicated in paragraph 3.1. Mechanical sieving is used to extract the shives after module 3. However, some shives remain even after mechanical sieving. These shives are manually extracted and their morphology analysed.

Table 5Shives granulometry for each module.

| | Average length (mm) | Average width (mm) | Average shape factor |
|-----------|---------------------|--------------------|----------------------|
| M1 | 7.27 | 2.17 | 4.16 |
| M2 | 7.46 | 2.64 | 3.56 |
| M3 | 6.48 | 2.27 | 3.25 |
| Sieved M3 | 13.32 | 3.70 | 3.93 |

They are represented in Fig. 6 as M3 sieving. The results obtained, presented in graph 2, show that the majority of shives extracted manually after sieving have lengths between 5 and 25 mm, and this in greater proportions than for un-sieved material. Lowest size shives with lengths between 0 and 5 mm are not very present. This suggests that sieving permits to remove from the lap the smallest shives. However, this is not sufficient to remove larger shives that remain entangled within the fibre lap. Different mechanical separation should be investigated and tested to better separate the remaining shives from the fibre lap.

3.2.5. Mechanical properties of the elementary fibres

Thirty elementary fibres are collected after each extraction stage in order to study the impact of each module (Module 1, Module 2 and Module 3) on the mechanical properties as well as on the number of defects (kink bands) in the elementary fibres at each extraction stage. In addition, individual fibres are taken from hemp stalks from the harvested batch, retted and baled. This is the same batch that is then processed with the Laroche Cadette 1000 fibre extraction device. Statistical studies are conducted using the raw material as a reference. The results obtained are presented in Table 6.

Placet et al. (2012) reported the results of different studies dealing with the properties of single hemp fibres. In some studies, the fibres are extracted manually and carefully directly on hemp stems (without any industrial mechanical processing) (Duval et al., 2011; Thygesen et al., 2007). The values given by Thygesen et al. (2007) show very high values of the tensile strength of Felina fibres (1735 MPa) but a relatively low elastic modulus (25 GPa). The high value of the tensile strength may be explained by the fact that the fibres were extracted manually. In those conditions, one can expect that the fibres were less damaged. This hypothesis is however not confirmed by Duval et al. (2011) who studied the importance of the location of the fibre in the plant length. In the best case they measured strength of about 480 MPa and a modulus of elasticity of 20 GPa for Fedora 17 manually extracted fibres. If the manual extraction method was similar, other factors

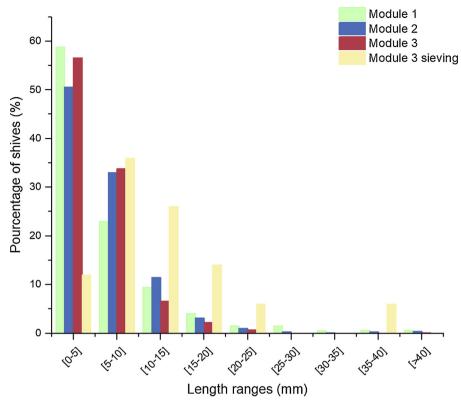


Fig. 6. Shives length distribution for each module.

Table 6Influence of extraction steps on mechanical properties.

| Batch | Failure stress (MPa) | Modulus of elasticity (GPa) | Number of kink bands (/330 μm) |
|----------------------|-------------------------|-----------------------------------|--------------------------------------|
| Raw material | 719 ± 495 | 42 ± 29 | 6 ± 4 |
| M1 | 660 ± 354 ^a | 38 ± 11 | 7 ± 4 |
| M2 | 620 ± 429 ^a | 37 ± 27 | 10 ± 4 |
| M3 | 528 ± 304^{b} | 37 ± 16 | 12 ± 5 |
| Placet et al. (2012) | 636 ± 253 | 25 ± 11 | n.a. |

Means in the same column with the same letter (a–b) are not significantly different at $P < 0.05; \, n.a., \, not$ available.

affected severely the strength of the fibres in comparison to the ones tested in (Thygesen et al., 2007). Marrot et al. (2013) also compared the mechanical performances of Fedora 17 hemp fibres manually extracted from two different locations in France. In the best case, the properties were improved both in strength (889 MPa) and modulus (35 GPa) in comparison to the ones measured by Duval et al. (2011). The authors of this study investigated the biochemical composition of the fibres and middle lamellas. They indicated that the biochemical composition could not explain the relatively low mechanical properties in comparison to flax for example. However, they found out that the middle lamellas were very strong and lignified as the stems were not retted. They therefore attributed the relatively low strength to the fact that the decortication, even if manually performed probably introduced weaknesses within the fibrous structure.

For stems processed by a "all fibre" equipment (hammer mill), Placet et al. (2012) presented properties for unknown hemp fibre variety from the LCDA (La Chanvrière De l'Aube company, France) with strength values of 636 MPa and modulus of elasticity of 24 GPa (Table 6). For these fibres one do not know if retting was performed. One can assume that it was performed following the requirements of the paper industry. The values presented in Placet at al. (2012) are for

strength in accordance to the ones presented in this work. However, the modulus of elasticity is lower than the ones measured in this study (37 GPa in the worst case).

The results presented in this study for mechanically extracted fibres are higher than the results presented in the literature for the modulus of elasticity (about 1.5 higher than for Placet et al. (2012) which was considered as a good value for mechanically extracted hemp fibres). It can be observed that a relatively low variation in modulus takes place between the fibres extracted manually and the fibres submitted to the three extraction modules. Non-significant decreases are observed between the modulus of the fibre manually extracted from the straw and the ones submitted to the three extraction modules. This therefore means that the internal structure of the fibre is probably not damaged to a large extent during the extraction. The failure stresses of the fibres extracted from the various modules varies from 660 to 528 MPa for M1 and M3 respectively. The values are highly scattered but Student tests show that Modules 1 and 2 do not differ significantly from the raw material. However, the fibres coming from M3 differ significantly from the raw material.

One can believe that the fibres were probably already damaged by the mechanical harvesting using a combine machine as the value of the mean tensile strength is much lower than the one presented by Thygessen et al. (2007) for manually cut stems and fibres extracted manually with care. However, the mechanical fibre extraction does not impact the modulus of elasticity. This probably indicates that the mechanical loads applied to the stems and to extract the fibres may have more effect on the strength than on the elastic modulus. This would explain the fact that our values of elastic modulus remain higher even after a relatively severe mechanical treatment than in other studies.

In Table 6, the levels of standard deviation are well within the classical ranges observed for such properties. They are actually reduced in comparison to other studies, because the authors used the Dia-Stron device for the fibre tensile tests. This device has for tendency to reduce the measurements errors usually performed when measuring the fibre diameter using the classical microscope method. This is actually

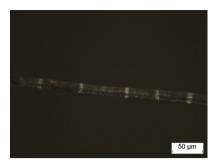


Fig. 7. Kink bands on hemp elementary fibre.

explained by Garat et al. The large distribution of values is generally due to the high variability of the natural fibre properties and also to some errors that can be systematically performed in some cases.

Table 6 also presents the number of kink band defects measured under polarized light (Fig. 7) as a function of the different extraction phases. The number of kink bands (~ 18/ mm) in the raw material is equivalent to the maximum number observed by Beaugrand et al. (2017) for processed fibres with a "all fibre" extraction line. In our case, the average number of kink bands after M3 is about 2.25 times larger. Table 6 indicates that defects were already present in fibres extracted from the mechanically harvested stems. This could explain the fact that the tensile strength of the fibres is much lower than the one presented in Thygesen et al. (2007). The number of defects raises for the different successive extraction modules but the difference is only statistically significant between the raw material and M3. This statement follows the one encountered for the decrease in tensile strength. One may be tempted to relate the increase in kink band defects to the decrease in tensile strength of the fibres but not to the elastic modulus.

The subject of the influence of the kink band defect on the tensile properties of natural fibres is treated by numerous authors as stated in the introduction section (Baley, 2004; Bos et al., 2002; Symington et al., 2011; Yan et al., 2014) as well as by Nilsson and Gustafsson (2007); Hanninen et al. (2011). In his review, Hughes (2012) explains that most authors conclude that the kink bands are places where strain concentration takes place. This should have for consequence to reduce the modulus of elasticity of the fibres. This however is not observed by all authors (Thygesen et al., 2007). Thygesen et al. (2007) shows that the increasing area of kink bands does not change the rigidity of the fibres and does not change the tensile strength. They also explain that the number, or surface of defects observed is not significant to explain a possible variation in mechanical properties because the kink bands can be visually suppressed by straining the fibre for example. However, removing visually the kink bands does not mean that the kink bands are not places of possible fibre weakness. Baley (2004) shows that the kink bands are probably crack initiating zones as they showed by observing the surface of the fibres during a tensile test by SEM. Beaugrand et al. (2017) more recently used X-ray micro tomography and show that the defect zone is a place that favours the initiation of crack throughout the thickness of the fibre. Baley (2004) however does not find a link between the number of defects and the strength despite the fact that the kink bands are privileged zones of crack initiation. This may be due to the fact that the fibres during their growth history as well as during their extraction from the stem (mechanically or manually) are submitted to bending solicitation (and therefore with one side of the fibre in compression) creating the kink band defects. Some strains applied during the fibre extraction may reduce the appearance of defects but not necessarily a possible micro-crack. Aslan et al. (2011) conclude that despite the fact the number of defect is not necessarily an indicator to predict the reduction of the fibre mechanical properties, they observed that the strength of flax fibres, between carefully manually extracted fibres and mechanically processed ones, is decreased by 44 %.

All the information provided in the last paragraphs indicates that

the modulus of elasticity may not be necessarily reduced by the fibre extraction processing as shown in this study. However, one may think that the modulus of the fibres may have been reduced during the harvesting and baling phases as defects are already present in these fibres in a larger quantity than what is usually observed (Beaugrand et al., 2017). The strength is decreased by the mechanical fibre extraction procedure used in this study when the fibres are submitted to the 3 modules. This type of observation was globally observed by all the previously cited authors and is due to the external loads brought to the stems to extract the fibres. With the view to maximise the strength of the fibres, one could recommend to process the fibres in only two extraction modules as the decrease in strength is not significant up to the second module and because the amount of fibre extracted is equivalent to the one obtained after module 3. The results obtained during this study indicate that it may not be necessary to extract the fibres using all the modules of the fibre extraction device. Indeed, even if module 3 offers the possibility to obtain higher fibre rates, it leads to a degradation of mechanical properties. A mechanical sieving step would therefore be required so that to extract a maximum amount of the remaining shives of the lap after module M1 or M2. It would therefore be interesting to stop fibre extraction after module 2 and not after module 1 since the fibre content remains the same and this allows the possibility to reduce the size of the shives and thus to extract them more easily by sieving.

The mechanical performance both in rigidity and in stress to failure indicate that the hemp fibres extracted using the all fibre opener described in this work can be considered for reinforcing polymers for composite materials. The modulus of elasticity (38 GPa) is sufficiently high to provide a good reinforcement to polymers which modulus do not overpass 3 GPa. The strength of the fibres is also in the top range of hemp fibres extracted mechanically and sometimes higher than in studies where hemp was manually extracted. This suggests that the hammer mills (Xu et al., 2012) or other aggressive extraction techniques such as the one used in (Placet et al., 2017) that provide fibres with poor properties should not be considered for other applications than paper or short fibre large diffusion composites.

Of course, the mechanical properties obtained in this work with hemp fibres should not be compared to the typical values encountered in the literature for scutched/hackled long flax fibres (Bourmaud et al., 2018) as the whole processing technique to extract the fibres is more rudimentary and is linked to the fact that in western Europe the hemp is harvested using a combine machine. To obtain higher fibre mechanical properties, one should probably adapt the traditional harvesting of hemp stems as performed in Eastern Europe by using harvesting devices such as the one described in Pari et al. (2015) for harvesting and designed by the Polish Institute of Natural Fibres & Medicinal Plants (IWNIRZ) that could separate the top of the stem from the bottom and lay the bottom part in an aligned way. After dew-retting, the stems could be processed in dedicated scutching/hackling industrial lines such as the ones presented by Turunen and Van Der Werf (2007) or in adapted flax lines so that to produce long fibres with probably even higher mechanical properties than the ones presented in this study.

As the fibres exit the "all fibre" extracting device entangled in the form of a mat, these ones need to be processed so that to transform them in 1D textile products such as yarns and architectured textiles. The length of the fibres (between 38 and 57 mm) depending on the extraction module M1 or M3 is sufficient for the carded spinning route. The main difficulty to achieve textile products (structural woven geotextiles or woven composite reinforcements) from the fibres extracted with the all fibre opener is to reduce the technical fibre diameter without using damaging chemicals so that to give more volume to the fibre and facilitate the carding step and the realisation of a carded web with enough cohesion. This point is under investigation and will be the subject of future works.

4. Conclusions

Mainly cultivated for its seeds, the hemp is harvested using devices that form a windrow composed of randomly aligned straws. Hammer mills, used to extract the fibres for low added value applications, damage the mechanical properties of the fibres. In this work another type of all-fibre extraction device (A laroche Cadette 1000 opener) is used with adjusted settings and procedures with the goal to keep the morphological and mechanical potential of hemp fibres as high as possible. The chemical, morphological and mechanical properties of the fibres is investigated. It is shown that the fibres extracted with the commercial extracting device have sufficient length to be further processed into technical textiles. The mechanical properties of the extracted fibres are situated in the higher range of hemp fibres mechanically extracted from the literature. The morphological and mechanical properties measured in this work indicate that the use of an all-fibre opener using rotating pin roller properly set-up opens the possibility to manufacture textile products by the carding route, for mid-range load-bearing co-mingled composite parts or into load-bearing geotextiles/geocomposite applications for example. This work demonstrates that an efficient extraction technique can be used industrially to obtain higher performance hemp fibres therefore opening and diversifying the application field for fibres that are generally considered for low added value applications because of the damages conferred to them by hammer mills. This may therefore open the possibility to value the hemp straw in a higher manner and this could have for effect to raise the level of cultivation of hemp in zones where large extraction facilities are not present because this type of device can be proposed with different sizes and could be used by local groups of farmers to extract themselves their fibres in territory where no large extraction facilities are present.

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