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Fibre Extraction from Oleaginous Flax for Technical Textile Applications: Influence of Pre-processing parameters on Fibre Extraction Yield, Size Distribution and Mechanical Properties

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Abstract

Cultivated primarily for its seeds, oleaginous flax could also be valued for the different fractions that can be extracted from the straw. However, as the straws are not harvested with the same technique and care than for the textile flax, the classical scutching technique cannot be used. As a consequence, an “all fibre” device was used to perform the separation of the different constituents of the oleaginous flax straws. The different fractions were quantified for two retting levels and for two degrees of rewetting of the stems. The physical and mechanical properties of fibres were then evaluated. It appears that the relative amount of fibres extracted from oleaginous flax straw is comparable to the one from textile flax (i.e. 40% of the stem dry mass), and their tensile properties are situated in the lower part of the textile flax range. This work shows that the individual fibre length of oleaginous flax (between 3 and 6 cm) is comparable to that of the scutched textile flax fibres. This makes them suitable for the production of carded aligned fibre yarns for technical reinforcement textiles (e.g. composites or geotextiles). These results demonstrate the interest and the potential added value of harvesting the stems for technical fibre applications.

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Keywords: Oleaginous flax, Fibre extraction, Extraction yield, Size distribution, Mechanical properties.

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1. Introduction

Oleaginous flax is cultivated primarily for its seeds which can contain up to 45% of vegetal oil. Linseed oil is used in many industrial sectors. Firstly, the major fatty acids of linseed oil are α -linolenic acid (C18:3 ω -3) (56%), linoleic acid (C18:2 n-6) (16%) and oleic acid (C18: 1 n-9) (18%). Thus, it is an important vegetal source of omega-3 and omega-6 fatty acids, and it is therefore widely recommended for human consumption. Secondly, thanks to its nourishing properties, linseed oil is also used for the formulation of many cosmetic products. Lastly, linseed oil is a drying oil which spontaneously cures in air. It is therefore frequently used as a siccativ for vegetable inks, as well as in the formulation of oil paints, floor cleaners and wood preservatives.

Shives, the ligneous part of the flax stems, can be extracted. They are mainly used as animal litters because of their high water absorbency. However, it is reasonable to assume that flax shives could be also used (i) as reinforcing fillers for the production of extrudable and/or injection-mouldable wood-polymer composites [1], (ii) for the manufacture of renewable thermal insulation or high-density fibreboards [2,3] using hot pressing, (iii) for the production of thermal insulation blocks using compression moulding [4], or (iv) as bio-aggregates to be mixed with mineral binders for the design of bio-based concretes [5]. On another side, vegetal dust, another part of the flax stem, could also potentially be used for the mechanical reinforcement of thermoplastic polymers in the plastic industry.

The last vegetal fraction of interest in oleaginous flax stems are the fibres. In a study published in 2011 by the French National Environment Agency (ADEME) [6], it appears that 11,000 ha of oleaginous flax are cultivated in France each year. This is a small fraction of the worldwide production which was identified in 2009 to be of about 1.7 Mha from which Canada is the main producer with 650,000 ha. The amount of straw is generally considered to be about 2 tons/ha for oleaginous flax. However, it has to be mentioned here that the size of the flax stem is prevented to grow so that to avoid falls of the plant in the field, thus associated to troubles for the harvesting. In their study reporting the influence of the genotype of eleven varieties, Rennebaum et al. [7] showed that up to 5.5 t/ha of straw can be harvested without compromising the amount of seeds. If seeds of oleaginous flax are well valued, the rest of the plant, and particularly the straw, is most of the time not used at all. It is shredded simultaneously during the seed harvesting. This explains mainly why technical textiles from oleaginous flax are not available on the market even if the mechanical properties of the linseed individual fibres reported in relatively few studies [7, 8] indicate that they are situated in the lower range of the textile flax fibre mechanical properties. Pillin et al. [9] showed that single oleaginous flax fibres manually extracted with care exhibit tensile properties that are situated within the range of properties measured for textile flax fibres also manually extracted, the latter being commonly used for the production of reinforcement textile for structural or semi-structural composite applications. However, this last statement may not be true anymore if one considers the properties of the fibres after all the mechanical extraction phase of the fibres from the stems.

The harvesting of oleaginous flax does not allow the straw to be processed with the same techniques than the ones used for textile flax. The straws are mown and directly absorbed by the combine harvesting machine which separates the seeds from the straws with its integrated threshers. The straws are therefore submitted to mechanical loadings during the beating phase. At the end of the threshing phase, the straws fall regularly from the combine harvesting machine and form a windrow of randomly oriented stems. The straws can then be left in the field so that a dew-retting performed by soil microorganisms can take place. This pre-processing stage, which is well known and documented for textile flax cannot be performed with the same protocol for linseed flax as the straws are not aligned and well distributed on the ground. During the dew-retting, the contact with the soil and therefore the microorganisms are not similar for all the pieces of straws. Even if it would be possible to return the windrow during time, the evenness of the dew-retting may be questionable. Moreover, as the fibres are not aligned within the windrow, the linseed flax stems are packed with random orientations in large balls each of about 200 kg, and the stems cannot be aligned as it is the case in the traditional scutching and hackling route usually employed to separate the different vegetal fractions of the plant for the textile flax. As a consequence, an "all fibre" device has to be used. Different devices, inspired from the paper industry are generally used. However, these devices are often very aggressive to the fibres, and they may lead to the appearance of defects such as dislocations within the fibres as it is the case during the extraction of hemp fibres [10].

The aim of this work is to study the impact of two pre-processing parameters (i.e. the degree of retting and the moisture content at the inlet of the fibre extraction machine) on the extraction yield of fibres from oleaginous flax

straw submitted to all the mechanical harvesting and extracting procedures, on their length, and on their tensile properties.

2. Materials and methods

2.1. Material

All trials were carried out using two different batches of oleaginous flax (*Linum usitatissimum* L.) straws (Everest variety), cultivated in the South West part of France and supplied by Ovalie Innovation (Auch, France). The first batch consists of straws collected and packed into balls of 200 kg immediately after the seed harvesting, i.e. at the beginning of July, thus corresponding to the non-retted batch (NR). The second batch (R) consists of straws retted during three weeks after the seed harvesting. Both batches originated from the same field.

Additionally, the two previously described batches were rewetted by sprinkling of liquid water before the extraction of fibres. A third and a fourth batch were then created. These are the wet non-retted (NRH) and the wet retted (RH) batches.

2.2. Analytical methods

As the mechanical behaviour of ligno-cellulosic fibres and their associated composites change as a function of their moisture content [11, 12], it is important to determine the level of absorbed water at the inlet of the fibre extraction device for all the considered batches. By doing so, it is possible to investigate if the humidity has an influence on the physical and mechanical properties targeted in this work. The moisture contents were determined according to ISO 665:2000 [13].

2.3. The fibre extraction device

A Laroche (France) Cadette 1000 “All Fibre” extraction equipment located in the AGROMAT platform (Tarbes, France), the technological transfer hall of Laboratoire de Chimie Agro-industrielle, was used to separate the different vegetal fractions from all the oleaginous flax straw batches considered in this work. A schematic diagram of the device is presented in Fig. 1.

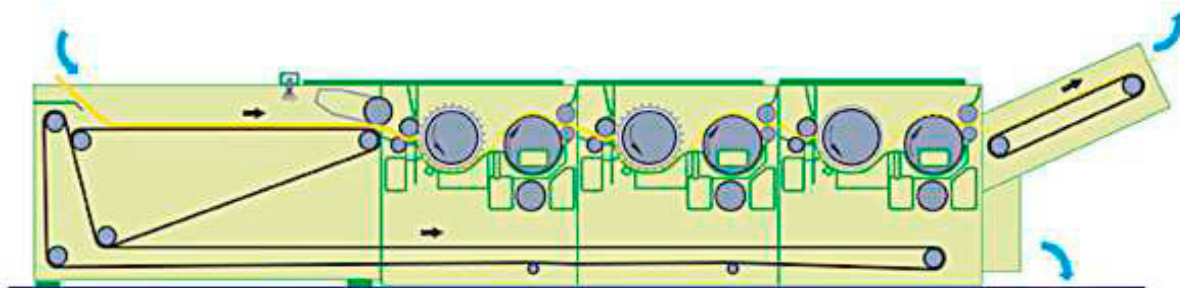


Fig. 1: Laroche (France) Cadette 1000 fibre extraction device (from Laroche company website)

With a 1 m width, this tearing machine has the capacity to both perform the opening and the cleaning of natural fibres, as well as the realization of laps. It is equipped with three modules. At the inlet of each of them, the feeding of the raw material is ensured by a pair of rollers, one smooth and the other grooved (made of rubber). Then, each module has a cylinder equipped with nails, i.e. the extracting roller (or fibre extraction roller). Its rotation speed is adjustable (from 750 to 1800 rpm). Under the extracting roller, a trap door allows the flax shives to be evacuated by gravity.

At the end of each module, there is also a perforated cylinder at which ventilation is applied. The perforated cylinder has three functions: extracting the vegetal dust from the material, forming the lap, and transferring it to the next module or the outlet. Each de-dusting fan is equipped with a motor with a maximum rotation speed of 2865

rpm.

During the experiments, the inlet flow rates of oleaginous flax straw were approximately 175 kg/h, corresponding to a 3.5 m/min feed belt speed. The transmission speed of the lap from module 1 to module 2 was 2.2 m/min, and it was 1.5 m/min from module 2 to module 3. Finally, the speed of the output belt was 1.8 m/min. The rotation speed of the extracting rollers was 725 rpm in each of the three modules. The rotation speed of the motor in the de-dusting fans was 1500 rpm for module 1, and 2000 rpm for the two next modules.

2.4. Production and vegetal fractions

All trials were carried out on retted (R) and non-retted batches (NR). From both batches, two experiments were conducted, one without rewetting (RS and NRS) and another with rewetting by liquid water (RH and NRH). Each sampling was carried out for 10 min. Then, the three fractions obtained (lap, flax shives, and dust collected from the three modules mixed in one only dust fraction) were weighed. The material balance can then be determined for each sampling. The lap consisted mainly in fibres but also in some flax shives trapped inside the lap. From each lap, a 20 g sample was collected to determine the content of flax shives inside the lap, and thus the real fibre content. A manually sieving was performed on the lap for 2 min in order to drop flax shives. Then, the residual flax shives still trapped were collected manually. Fig.2 shows photographs of the three obtained vegetal fractions.

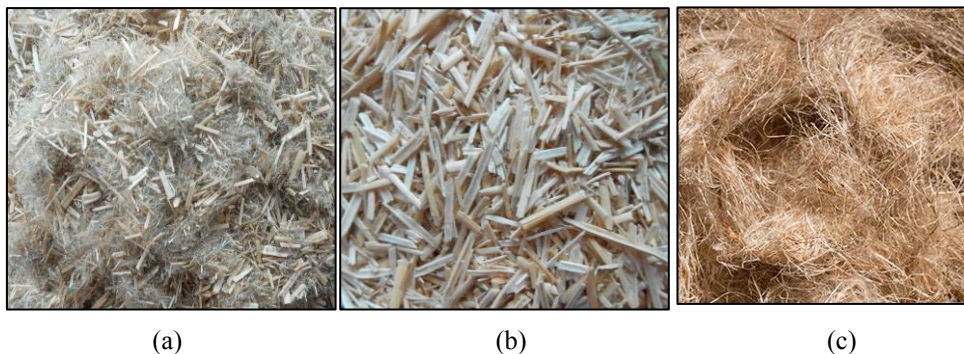


Fig. 2. Photographs of (a) vegetal dust, (b) shives, and (c) Technical fibres after all the extraction operations

2.5. Fibre length

From laps without residual flax shives, a handful of fibre bundles was extracted and 500 bundles were collected. Every fibre bundle was measured in length. One extremity of the fibre bundle was fixed and the other was pulled to extend the fibre bundle. The length was measured between these two extremities. The arithmetic mean value of the length was then calculated as well as the corresponding standard deviation.

2.6. Tensile testing on elementary fibres

For each batch, between 24 and 30 thirty elementary fibres were manually extracted from fibre bundles. With the aim of facilitating the extraction of elementary fibres, fibre bundles were dipped in boiled water for 20 min. According to Charlet [14], this pre-treatment does not have any influence on the tensile properties (i.e. tensile strength and Young's modulus) of the flax fibres. Elementary fibres were glued on a paper frame to have a gauge of 10 mm, due to the short elementary fibre length (from 10 to 50 mm). Following this step, fibres were stored indoor. They exchanged moisture with ambient air and thus dried until reaching a moisture content corresponding to their equilibrium with the surrounding atmosphere ($T = 21^{\circ}\text{C}$, $\text{RH} = 65\%$). Before tensile test, the average apparent diameter of each fibre was determined. It is an average value from five points measured along the elementary fibre using an optical microscope (Olympus PMG3-F3, France). To calculate the tensile Young's modulus and strength, the effective cross-sectional area was calculated from this average apparent diameter, considering the fibre as perfectly cylindrical. The paper frames were clamped on a Bose (USA) Electroforce 3230 tensile testing machine

equipped with a 22 N capacity load cell. The tensile properties were then determined in accordance to the appropriate standard test method [15].

3. Results

3.1. Analysis of the vegetal fractions

As explained in Sections 2.3 and 2.4, the Laroche Cadette 1000 fibre extraction device offers the possibility to separate (at least partially) the different fractions of fibre plants such as the oleaginous flax stems. The vegetal dusts and the shives are both sent to reception bags via pneumatic systems, and the fibre laps go to the machine output belt. The different amounts of the three extracted vegetal fractions are presented after one extraction cycle through the three modules of the device in Table 1. The experimental results given correspond to the different matters collected at the outlets of the extraction device for 100 kg dry matter oleaginous flax straw at the inlet. The amount of humidity is therefore subtracted from the masses of the different constituents. Moisture contents of the four considered batches, i.e. RS, NRS, RH and NRH, are 8.7%, 9.1%, 16.0% and 18.1%, respectively.

Table 1: Fractions collected at the outlets (dry weights expressed for 100 kg dry matter at the inlet).

	Straw (kg)	Fibre Lap (kg)	Shives (kg)	Dust (kg)
RH	100.0	54.0	40.8	5.2
NRH	100.0	56.0	38.3	5.7
RS	100.0	66.6	23.6	9.8
NRS	100.0	57.3	37.2	5.5

Results presented in Table 1 show that the amounts of shives extracted from the re-wetted batches are much greater than from batches simply stored at ambient temperature. The retting also favours the extraction of shives from the re-wetted batches. For the ambient humidity batches, the retting has for consequence to favour the extraction of dusts but not the extraction of shives.

On all the four batches, visible amounts of shives are still part of the fibre laps. This is the reason why an additional manual sieving step followed by a manual collection of the last pieces of shives was performed to quantify the amounts of shives left into the fibre laps. The results are presented in Table 2.

Table 2: Mass fraction of shives within the fibre laps after one extraction processing step.

	Fibre content inside lap (%)	Shive content inside lap (%)
RH	69.3	30.7
NRH	72.1	27.9
RS	56.7	43.3
NRS	68.4	31.6

Table 2 indicates that for three of the four batches, i.e. both re-wetted batches (RH and NRH) and the non-retted batch from ambient condition storage (NRS), the amount of fibres within the laps is about 70%. For the RS batch, more shives are trapped within the laps as 43% of the lap mass is constituted of shives. These results clearly indicate that the laps need to be further processed to remove residual shives, either by using a sieving step or by submitting the laps to a second step into the fibre extraction equipment.

After the full separation of shives and the removal of as much vegetal dust as possible, the final amounts of fibres, shives and dusts are showed in Table 3:

Table 3: The three vegetal fractions after full separation of the constituents (results are given in mass percentage).

	Total fibre content (%)	Total shives content (%)	Total dust content (%)
RH	37.5	57.6	4.9
NRH	40.4	53.9	5.7
RS	37.8	52.4	9.8
NRS	39.2	55.3	5.5

Table 3 indicates for the four considered batches the respective amounts of the extracted constituents from the oleaginous flax stems. More than half of the stem mass is constituted by shives. The values are situated in a 52-58% range, depending on the initial pre-processing treatment imposed to the stems. One can assume that the result dispersion may be relatively large because these ones may depend on the way to select the stems in the balls and on the exact degree of shive extraction from the lap. As the criterion is visual, some variations may happen and some small pieces of shives or dust particles may still be part of the laps even if this is in a very small proportion.

The proportion of fibres extracted from the stem is situated in the 38-40% range. The influence of retting and humidity do not have particular influence on the linseed fibre yield. This therefore suggests that the pre-processing treatments are not influential if the degree of fibre extraction is the only considered point of view. For the RS batch, the amount of dust is larger than for the three other batches. This may be due to the fact that during the fibre extraction, some pieces of fibres and/or shives are cut and transformed into very small particles (i.e. dust) whereas for the other pre-processing conditions, more dust is left within the fibre laps or around the shives.

The proportion of fibres extracted from the oleaginous flax stems (38-40%) is very much larger than what was reported in the very few studies found in the literature. In a study from the French National Environment Agency (ADEME) [6], the respective amounts of fibre, shives and dust were 25%, 65% and 10%. In their study, Rennebaum et al. [7] found an average fibre yield of 23% for eleven different varieties with a peak value at 29%. Our results therefore show that a greater proportion of fibres are extracted, in combination with a lower amount of shives (the dust proportion being quite equivalent in all studies). One could argue that the laps considered in this study still contain some shives or dusts. But, this is not the case because shives were at the end of the process removed by shaking and sieving, and the last pieces of shives were carefully extracted by hand with a pair of surgical tweezers. The global amount of fibre extracted from the oleaginous stems is comparable to the percentage of fibre extracted from textile flax (i.e. 25% of long fibres plus 15% of short fibres (tows), corresponding to a 40% total amount of fibres) [6].

The results presented in this study indicate that with the stems from the Everest variety, cultivated and harvested by Ovalie Innovation for their seeds, the relative percentage of fibres that can be extracted from oleaginous flax is much higher than for the previous studies. This large proportion of fibre could increase the economic interest of harvesting the flax stems instead of shredding them during the seed harvesting.

3.2 Fibre properties

The mean fibre lengths from the four different batches studied in this work are presented in Table 4.

Table 4: Length of technical fibres.

	Fibre length (cm)
RH	5.3 ± 2.9
NRH	5.1 ± 2.9
RS	3.9 ± 2.2
NRS	3.7 ± 1.9

The results presented in Table 4 show that the re-wetting pre-processing parameter has for consequence to keep the length of the technical fibres. The retting does not have any influence on the fibre length. When re-wetting the fibres at the inlet of extraction device, their length remains larger (5 cm instead of 4 cm without re-wetting). This is due to the decrease in the fibre rigidity thus leading to less breakages.

Table 5: Diameter and tensile strength of individual fibres.

	Diameter (μm)	Tensile strength (MPa)	Maximum strength (MPa)	Minimum strength (MPa)
RH	22.2 \pm 3.7	377 \pm 189	980	153
NRH	21.3 \pm 3.9	371 \pm 160	774	105
RS	23.7 \pm 5.2	324 \pm 110	578	115
NRS	20.2 \pm 4.3	333 \pm 108	635	167

The results presented in Table 5 show that the diameters of the individual fibres (20–23 μm) are well situated in the range already observed in previous studies for textile and oleaginous flax [7–8]. The values of the tensile strength are situated for the four studied batches in the 333–377 MPa range. If one can observe that the values from the re-wetted batches show larger values, these ones are not however statistically different (student test) than the two others. It is therefore difficult to conclude on the interest of any pre-processing treatment concerning the mechanical properties. The values presented in Table 5 are very much comparable to the ones presented by Remebaum et al. or Tomljenovic et al. [7,8] (average values of about 420 MPa). In both studies, they used mechanical extraction processes to extract the fibres from the stems after a one-month dew-retting. On the contrary, Pillin et al. [9] extracted the fibres manually directly from the stem. Their results show much higher tensile strengths (863 \pm 444 MPa). The dispersion of results observed in this study is also very large as shown by Table 5. The highest results are well situated in a range of values presented by Pillin et al. [9] whereas the lowest values are relatively low. This is probably due to the fact that the fibres they used were not submitted to any mechanical stress to separate the fibres from the shives and dusts. Defects such as kinks or dislocations are probably introduced during the fibre extraction steps as it was previously observed for the fibre extraction from hemp [10] with similarly aggressive extraction techniques. Some fibres are probably more affected by the fibre extraction process than others. The coefficient of variation for the re-wetted fibres 0.5 and 0.43 for RH and NRH are larger than for the two other batches (0.33 and 0.32 for RS and NRS). For the RS and NRS batches, the coefficients of variation are lower than for the re-wetted batches. This may suggest that extraction defects systematically take place in the RS and NRS batches whereas for the RH and NRH batches higher maximum strength values are observed for some fibres (980 MPa for RH) suggesting that some of them were extracted without any damage. In this case, the value of the maximum tensile strength observed for the RH and NRH batches in this work are within the range of properties of fibres extracted manually [9, 16] without defects.

Limitation of defects by optimising the extraction parameters or by using softer extraction techniques should be considered in future works. However, even if the tensile strength is affected by the extraction process and probably reduces the potential properties of fibres by about half, these ones show values that are still large enough for applications in technical textiles for semi-structural composite parts or for geotextiles.

4. Conclusions

The fibre extraction from oleaginous flax straw and particularly the influence of pre-processing parameters such as dew-retting and re-wetting of stems before extraction was investigated in this work. The results indicate that the retting considered in this study globally does not have any influence on the extracted fractions of fibres and shives. More studies should be performed to confirm this result with other retting conditions. The re-wetting of the stems

prior to the fibre extraction has for tendency to favour the extraction of shives. The amount of fibres extracted from the flax stems studied in this work is much larger than what was found in previous studies. It is difficult to explain this very attractive result (obtained for all the studied batches) at this stage, but this may be attributed to very good growth conditions associated to a well-adapted extraction process which well keeps the fibres together and does not break a relatively large part of them. Further studies should be performed with other batches of stems of different varieties because if the results obtained in this study were confirmed, this would certainly increase the economic potential of oleaginous flax as the straw could be more valued than it is at the present time. The mechanical potential of the fibres was shown to be affected and therefore reduced by about 50% of its initial potential evaluated from fibres manually extracted with the greatest care [9], but the properties remained at a level that is completely acceptable for technical applications such as semi-structural composite parts or geotextiles.

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References

- [1] G. Gamon, Ph. Evon, L. Rigal, Twin-screw extrusion impact on natural fibre morphology and material properties in poly(lactic acid) based biocomposites, *Ind. Crops Prod.* 46 (2013) 173-185.
- [2] Ph. Evon, V. Vandenbossche, P.Y. Pontalier, L. Rigal, New thermal insulation fibreboards from cake generated during biorefinery of sunflower whole plant in a twin-screw extruder, *Ind. Crops Prod.* 52 (2014) 354-362.
- [3] Ph. Evon, J. Vinet, L. Labonne, L., Rigal, Influence of thermo-pressing conditions on mechanical properties of biodegradable fibreboards made from a deoiled sunflower cake, *Ind. Crops Prod.* 65 (2015) 117-126.
- [4] Ph. Evon, J. Vinet, M. Rigal, L. Labonne, V. Vandenbossche, L. Rigal, New insulation fibreboards from sunflower cake with improved thermal and mechanical properties, *J. Agric. Stud.* 3(2) (2015) 194-211.
- [5] E. Gazagnes, C. Magniont, G. Escadeillas, Matériau composite de construction incorporant de la chènevotte de chanvre. Brevet FR 2.946.640 (2009).
- [6] ADEME Report: Assessment of natural fibre availability in France. 2011. http://www.ademe.fr/sites/default/files/assets/documents/76290_12_evaluation_dispo_accessibilite_fibres_veg_usages_materiau_x.pdf
- [7] H. Rennebaum, E. Grimm, K. Warnstorff, W. Diepenbrock, Fibre quality of linseed (*Linum usitatissimum* L.) and the assessment of genotypes for use of fibres as a by-product, *Ind. Crops Prod.* 16 (2002) 201-215.
- [8] A. Tomljenovic, M. Erceg, Characterisation of Textile and Oleaginous Flax Fibrous and Shives Material as Potential Reinforcement for Polymer Composites, *Tekstilec* 59(3) (2016) 350-366.
- [9] I. Pillin, A. Kervoelen, A. Bourmaud, J. Goimard, N. Montrelay, C. Baley. Could oleaginous flax fibers be used as reinforcement for polymers? *Ind. Crops Prod.* 34 (2011) 1556-1563.
- [10] A. Hernandez-Estrada, H.J. Gusovius, J. Müssig, M. Hughes, Assessing the susceptibility of hemp fibre to the formation of dislocations during processing, *Ind. Crops Prod.* 85 (2016) 382-388.
- [11] V. Placet, O. Cisse, M.L. Boubakar, Influence of environmental relative humidity on the tensile and rotational behaviour of hemp fibres, *J. Mater. Sci.* 47 (7) (2012) 3435-46.
- [12] J. Moothoo, P. Ouagne, S. Allaoui, D. Soulat, Vegetal fibre composites for semi-structural applications in the medical environment, *J. Reinf. Plast. Comp.* 33 (2014) 1823-1834.
- [13] ISO 665: 2000. Oilseeds - Determination of moisture and volatile matter content; International Organization for Standardization: Switzerland, 2000.
- [14] K. Charlet, Contribution à l'étude de composites unidirectionnels renforcés par fibres de lin: relations entre la microstructure de la fibre et ses propriétés mécaniques. Thèse de Doctorat, Université de Caen, France (2008).
- [15] ASTM D3822 / D3822M – 14. Standard Test Method for Tensile Properties of Single Textile Fibers.
- [16] A. Lefeuvre, A. Bourmaud, C. Morvan, C. Baley. Elementary flax fibre tensile properties: Correlation between stress-strain behaviour and fibre composition. *Industrial Crops and Products* 52 (2014) 762– 769.