Industrial Crops & Products 170 (2021) 113736

Contents lists available at ScienceDirect

Industrial Crops & Products

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

Variety and growing condition effect on the yield and tensile strength of flax fibers

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A R T I C L E I N F O

Keywords:
Flax fibers
Varietal selection
Yield
Retting
Tensile strength
Natural fiber composites

A B S T R A C T

Flax (*Linum Usitatissimum L.*) fiber composites serve as sustainable replacement for glass fiber composites. Given that these composites are based on natural sources, their performance is susceptible to variation induced by several factors. Hence, ten flax fiber varieties cultivated in two different regions of Belgium in 2017 and 2018 are compared in terms of their yield and their tensile strength. Statistical analyses are carried out to determine the varietal difference in terms of tensile strength of bundles and of composites. The mechanical tests of flax fibers are carried out in two ways, i.e. dry fiber bundle tests and impregnated fiber bundle tests (IFBT). The results from the two consecutive growth years show their significant influence on the yield of the flax fibers, which are highly dependent on the weather conditions, in particular the precipitation during the growing season. The influence of the date of pulling and the retting degree on the yield of flax fibers and the tensile strength of fiber bundles is also investigated. The early pulling and lower retting degree result in a detrimental effect on the fiber strength whereas the late pulling shows little to no effect on the fiber strength. The difference of flax fiber strength among ten flax varieties over two years can be easily identified using IFBT and a couple of flax varieties exhibit a good tensile strength with low variation. This methodology can be used to select good flax varieties which have high mechanical properties with a small variation, which are suitable for a fiber reinforcement of high performance natural fiber reinforced composites.

1. Introduction

Over the past several decades, the usage of bio-sourced materials in the industrial sectors has steadily increased to decrease the environmental footprint, thus contributing to eco-friendly development. This increased usage has led to a great interest in academic and industrial research groups to provide bio-sourced materials whose properties can match the properties of existing petroleum-sourced materials. For example, composite materials, which mostly consist of petroleum-sourced polymers and synthetic fibers, are rapidly being replaced by biopolymers and plant-based fibers (CoDyre et al., 2018; Gurunathan et al., 2015). Nevertheless, bio-sourced materials still have some issues for the industrial applications, such as high variation of their properties due to their inherent nature of living organisms as well as low performance in terms of durability (Mak and Fam, 2020, 2019a; Pisupati et al., 2021) compared with the conventional synthetic materials. Therefore, the analysis of variation of their properties is one of key issues for the development of industrial applications using biosourced materials (Gurunathan et al., 2015; Mak and Fam, 2019b).

Among bio-sourced materials, flax fibers is an economically viable resource because it produces both fibers and seeds, which can be used for different applications (CoDyre et al., 2018; Khalfallah et al., 2014; Kim and Park, 2017; Pil et al., 2016). Flax plants contain long fibers which are distributed in the outer tissues of the stem in the form of bundles. These fibers can be obtained from the plant by following a series of successive steps such as retting, scutching and hackling (Van de Weyenberg et al., 2003). It has been reported that the quality of flax fibers is highly dependent on the careful execution of the aforementioned steps.

Furthermore, the quality and yield of flax fibers are also dependent on the growing conditions and varietal selection. The growth of flax plant is usually monitored in terms of accumulated temperature or effective cumulative temperature (Bert, 2013). It was identified that the optimum fiber richness is reached around an accumulated temperature

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https://doi.org/10.1016/j.indcrop.2021.113736
Received 22 February 2021; Received in revised form 7 June 2021; Accepted 8 June 2021
Available online 16 June 2021

of 850−1100 °C which is a favorable condition for harvest (Arvalis (France), n.d.; Bert, 2013). Higher accumulated temperatures (> 1100 °C) were found to be ineffective for the improvement of fiber richness. Higher accumulated temperatures cause lignin generation within the plant, however, and pose problems during the retting and mechanical separation of fibers (Bert, 2013). Meanwhile, the annual precipitation also influences the growth of fibers. It has been suggested that during the growing period, the precipitation should be about 110−150 mm (Heller et al., 2015). The relation between the precipitation and the tensile strength of flax fiber have seldom been reported in the literature. In the case of hemp fibers, however, about 10 % difference in average tensile strength was identified between the fibers cultivated in drought and in normal conditions (Thygesen and Asgharipour, 2008). This influence from the weather conditions can be reduced by cultivating flax in a greenhouse (Goudenhooft et al., 2018), which would lead to an increase in the overall cost of fibers.

Once the plants are harvested, the retting of flax stems is the first and one of the most important steps. This step is conventionally carried out in the fields where the harvested flax stems/straws are laid allowing the microorganisms in the soil to interact with the stems and to degrade the pectin from the middle lamellae. This facilitates the release of fibers from surrounding cortical tissues. This process is highly dependent on the weather conditions and is restricted to a geographical location with an appropriate climate, thus hindering the wider implementation. Hence, alternative solutions such as chemical and enzymatic treatments which replicate the natural process (Chabbert et al., 2020) can be introduced.

Although the use of enzymes improves the quality of fibers, these enzymes are too costly to replace dew retting (De Prez et al., 2018). Therefore, the dew retting which is highly variable, has become the only option in practice and its optimal duration needs to be determined. The quality of the fibers is dependent not only on the retting method but also on the retting duration. The fibers should be subject to retting for a specific duration to ensure a good retting degree while facilitating the separation of fibers from stems with less damage. If the fibers are retted for a longer duration, fiber properties can be degraded due to long time exposure to microbial activity. On the other hand, if the fibers are under retted, the fibers can be damaged further during the mechanical separation, leading to lower mechanical properties (Sisti et al., 2016). There is no standard approach for determining the optimal retting degree of flax fibers which has been mostly determined by a farmer's intuition and experience. Methods like thermogravimetry (Placet et al., 2017), biochemical analysis (Chabbert et al., 2020), or infrared analysis are adopted to identify the effects of retting duration at the lab scale. Nevertheless, these methods are rarely applied at large scales. Martin et al. (Martin et al., 2013) showed that a retting duration of 19 days increased the tensile properties of flax fibers by nearly 30 %. Bourmaud et al. (Bourmaud et al., 2019) has identified that the increase in the mechanical properties of flax fibers comes from the contribution of cell wall stiffening which is dependent on the retting degree and thereby suggesting a duration of about 20 days to be optimum.

On the other hand, the varietal selection of flax fibers is also a disputed topic. Some researchers suggest that the influence from the flax varietal selection on the mechanical properties of flax fiber reinforced composites is limited (Baley et al., 2019; Goudenhooft et al., 2019a, 2017; Lefeuvre et al., 2014; Pillin et al., 2011; Thuault et al., 2013) whereas others contradict that statement (Haag et al., 2017; Haag and Müssig, 2016). It should be noted that the properties of flax fibers are dependent not solely on their processing steps but also on their inherent variation because of different chemical compositions from variety to variety (Lefeuvre et al., 2015b, 2014).

The contribution of variety was found to be inconclusive, whereas it has rarely been examined in the literature from the point of view of composite properties (Baley et al., 2019). It has been shown that the varietal selection is important in terms of improving fiber yield whereas it has very small influence on the mechanical properties of fibers (Goudenhooft et al., 2017). In most of research work, however, the mechanical properties of fibers were characterized on a single fiber and their contribution to the composite properties was overlooked. Haag et al. (Haag et al., 2017) investigated the dependence of the composite properties on the flax variety and pointed out that the variety of flax is a factor to consider if the reinforcement is solely composed of a single variety of flax. The concept of varietal selection is also helpful to select disease tolerant crops which would result in a higher yield. In most of the studies, only a handful of varieties have been investigated (Pillin et al., 2011). In recent years, many new varieties of flax fibers are introduced into the market, which are claimed to be more disease resistant and/or give a higher yield. With the current growing needs and a potential change in the automobile sector, the demand for flax fibers can grow exponentially, which would call for higher yield crops. Thus, an investigation of these new varieties and their applicability to composite materials is necessary.

Hence, in this work, the scutched fibers of different varieties of flax fibers cultivated in two different regions in Belgium over a period of two years (i.e. 2017 and 2018) are investigated. The characterization of flax fibers is carried out from fiber scale to composite scale in order to investigate the influence of variety, the cultivation region and the year-to-year variation of weather condition on the yield and the mechanical strength of flax fibers. Furthermore, the influence of harvest time and duration of retting is also investigated. In particular, the tensile strength of flax fibers from different varieties are characterized using impregnated fiber bundle tests to select an optimal flax variety (Bensadoun et al., 2017; De Prez et al., 2019; Pisupati et al., 2019; Prapavesis et al., 2020).

2. Materials and methods

Ten different varieties of flax fibers registered within the European Union were cultivated in the regions of Rutten and Houtem in Belgium. The details of the plants and genus are presented in Table 1. Similar sowing densities (about 2000 germinable seeds/sq.m) were maintained for both fields to ensure a fair comparison between fields and varieties (Goudenhooft et al., 2017). The field tests were carried out for two consecutive years, 2017 and 2018. The flax plants were harvested after about 12 weeks and were dew retted for a period of about four weeks (see Table 2).

The monthly temperatures and precipitations for two consecutive years (i.e. 2017 and 2018) in both fields are presented in Table 3. The plants were pulled after about 100 days of sowing. Usually, the accumulated temperature received by the plants is used to estimate the harvest time of flax, which lies in the range of 850 ~ 1100 °C (Arvalis (France), n.d.; Goudenhooft et al., 2017; Pillin et al., 2011). It is calculated using Eq. (1).

$$\theta_{acc} = \sum_{i=1}^{n}\left(\frac{\theta_{max,i}+\theta_{min,i}}{2} - 5\right) \quad (1)$$

Table 1
List of flax varieties and growers of flax fibers.

Flax varieties	Grower	Year of inclusion in variety list
Aramis	SCA Terre de Lin, France	2011
Avian	Wiersum Plantbreeding,Netherlands	2013
Bolchoï	SCA Terre de Lin, France	2014
Calista	Groupe Limagrain, Netherlands	2009
Damara	Groupe Limagrain, Netherlands	2011
Elïxir	SCA Terre de Lin, France	2017
Novea	GIE Linéa Semences de Lin, France	2014
Nathalie	Van de Bilt zaden en vlas, BV, Netherlands	2013
Melina	Groupe Limagrain, Netherlands	2003
Vesta	Groupe Limagrain, Netherlands	2007

Table 2
Sowing and pulling dates for two trial years of 2017 and 2018.

	Houtem		Rutten	
	2017	2018	2017	2018
Sowing	30 March	20 April	25 March	14 April
Pulling	03 July	14 July	10 July	15 July
Pickup from field	28 July	16 August	27 July	22 August

where $\theta_{max,i,}$ and $\theta_{min,i}$ are the maximum and minimum recorded temperature on the *i*th day, respectively ($i = 1$ corresponds to the sowing date and $i = n$ corresponds to the pulling date). It should be noted in Equaton 1 that 5 °C is the lowest temperature above which the growth of flax is positive. Below 5 °C, the flaw growth is zero which corresponds to zero vegeration. Above 5 °C, the speed of development of flax is directly proportional to the sum of temperatures received by the plants since sowing.

The accumulated temperatures for the harvests in Houtem were 855.9 °C and 907.1 °C for the years 2017 and 2018, respectively. In Rutten, the accumulated temperatures were 1021.8 °C and 1138.4 °C for the years 2017 and 2018, respectively.

At another trial location, in the region of Beitem (Belgium), the influence of the date of pulling (early, normal, late) and the degree of retting on the quality of the flax fibers was investigated (see Table 4). This field test was also carried out for the same two consecutive years, 2017 and 2018. The accumulated temperatures in 2017 were 755.5 °C, 888.0 °C, and 1130.3 °C for early, normal, and late pulling, respectively. In 2018, they were 834.8 °C, 972.4 °C, and 1168.6 °C for early, normal, and late pulling, respectively. This trial was performed on the varieties of Vesta in 2017 and Calista in 2018.

2.1. Yield of flax fibers

The flax in the variety trials was grown on four repetitions with each surface of 20 m^2 at each field (in Rutten and Houtem). The flax was pulled at the same date for all the varieties with a flax pulling machine. The schedule of sowing and pulling is presented in Table 2. The monthly temperature and precipitation in Rutten and Houtem for the two consecutive cultivation years (i.e. 2017 and 2018) are presented in Table 3. Anyhow, it should be kept in mind that the fields in 2017 and the fields in 2018 were difference even at the same region (Rutten ot Houtem). Because a crop rotation is needed on the field and it is not recommended to cultivate flax for two consecutive years on the same field which causes problems with diseases. The fields in 2018 were nearby the fields in 2017, however and were in the same region with the same soil type. After the retting, the flax straw of each plot was manually bundled together. This flax straw was weighed to evaluate the yield of the retted flax straw. Afterward, this flax was scutched using a scutching turbine and the yield of fibers was obtained in terms of fiber mass per field surface. Among the fibers, only long fibers whose length was between 60 and 90 cm were considered to evaluate the yield of long fibers.

At the trial location in Beitem, at three different dates (early, normal, late), flax (Vesta in 2017 and Calista in 2018) on a surface of about 100 m^2 was pulled. Each surface of 100 m^2 was divided into three parts: a part with a short retting period, a part with a normal retting period, and one with a long retting period (see Table 4).

2.2. Measurement of fiber diameters

At least 40 fibers from each variety from each trial were carefully mounted onto an adhesive tape, which held them in place. The adhesive tape with fibers was later transferred into a gold coating chamber to facilitate microscopic observations using a Scanning Electron Microscope (SEM). SEM micrographs based on secondary electrons were acquired using an SEM (Joel Neoscope, 6000, Japan) with an applied probe current of 15 kV. The diameter of each fiber was measured at three different positions along the length of the fiber, and the average was reported as the fiber diameter. This procedure was repeated for all the fibers from each variety to report the average values and their distributions.

Table 3
Monthly maximum (T_{max}) and minimum (T_{min}) temperature and precipitation (Prp) in 2017 and in 2018 in Houtem and in Rutten.

Houtem							Rutten					
	2017			2018			2017			2018		
	T_{max} (°C)	T_{min} (°C)	Prp (mm)	T_{max} (°C)	T_{min} (°C)	Prp (mm)	T_{max} (°C)	T_{min} (°C)	Prp (mm)	T_{max} (°C)	T_{min} (°C)	Prp (mm)
March	13.53	5.00	9.20	–	–	–	13.88	4.49	11.80	8.72	1.35	63.80
April	17.70	3.84	15.00	16.41	6.64	87.60	14.05	2.42	22.20	18.79	7.12	50.40
May	19.87	8.93	29.60	19.31	8.22	12.40	22.61	8.93	33.40	24.55	9.22	22.40
June	23.85	12.65	8.40	20.84	10.35	11.80	25.37	12.44	52.80	24.49	11.93	71.40
July	23.35	12.65	68.40	25.77	14.09	28.20	24.03	12.98	81.40	30.47	13.13	4.60
August	22.94	12.07	138.80	23.01	13.27	91.00	–	–	–	–	–	–

Table 4
Dates of pulling and harvest, and retting degree for Beitem trials (a) in 2017 and (b) in 2018.

Date of pulling (2017)	Date of taking the flax inside after the retting (2017)	Retting duration (days)	Date of pulling (2018)	Date of taking the flax inside after the retting (2018)	Retting duration (days)	Retting degree	Reference
26 June	1 August	36	3 July	17 August	45	just scutchable	1-E
	8 August	43		28 August	56	good retted	2-E
	28 August	63		14 August	73	highly retted	3-E
06 July	8 August	33	12 July	17 August	36	just scutchable	1-N
	16 August	41		28 August	47	good retted	2-N
	28 August	53		14 September	64	highly retted	3-N
24 July	16 August	23	24 July	17 August	24	just scutchable	1-L
	22 August	29		28 August	35	good retted	2-L
	12 August	50		14 September	52	highly retted	3-L

2.3. Tensile test of dry fiber bundles

Initially, in order to characterize the tensile properties of the scutched fibers, flax fiber bundles were submitted to tensile load with a longitudinal displacement of 20 mm/min using a universal testing machine (Instron 5500R). These tests were conducted based on ISO 2062. Firstly, the scutched fiber bundles with an average linear density of 1500 tex were mounted onto a paper frame to facilitate the testing. The samples had a gauge length of 100 mm. The maximum force experienced by the fiber bundle divided by the linear density of the bundle was labeled as the fiber bundle strength (unit: cN/tex). In order to ensure repeatability, the tests were repeated at least 15 times to report the average values.

2.4. Manufacturing of composites

Quasi unidirectional composites were manufactured using hand layup followed by compression molding, whose method is similar to the one described in the literature (Bensadoun et al., 2017; Pisupati et al., 2019). A rectangular mold whose planar dimensions of 260 × 10 mm^2 was used. The high aspect ratio of 26:1 (i.e. length/width) was adopted for the mold dimensions because of two factors, viz. the arrangement of fibers and the test standard. A wide mold can allow to manufacture a large plate from which specimens for tensile tests can be extracted. In the current case, the fibers can neither self-align in one direction nor stitched. Hence, a mold with a higher aspect ratio will ensure the majority of fibers to be oriented in the longitudinal direction. The schematic of compression molding is presented in Fig. 1.

The scutched fibers were placed into the rectangular mold. Degassed resin-hardener mixture (Prime 27/Prime 20, Gurit, France) was poured onto the flax fibers. The excess resin was manually squeezed out using a roller to ensure no backpressure during compression molding. This step also reduces the residual void content within the composite (Bensadoun et al., 2017; Pisupati et al., 2019). The composite was cured at 60 °C for two hours under a compaction pressure of five bar and later transferred to a preheated oven for post-curing for seven hours at 65 °C to ensure complete polymerization as indicated on the technical data sheet of the resin (Gurit, n.d.). Considering the suggestions made by Haag et al. (Haag et al., 2017), the fiber volume fraction was maintained above 45 % for each composite specimen with an average thickness of 2 mm.

2.5. Tensile test of composites and IFBT

The composite specimens were cut to the dimensions of 250 mm in length to facilitate tensile testing according to the standard ISO 527. It is known that the properties of the flax fiber composites can be greatly influenced by the humidity (Mak et al., 2016; Mak and Fam, 2020). For this specific reason, the samples are stored in a controlled environment of 23 °C with 50 %RH until testing. The crosshead speed was set to 2 mm/min, and the strain was monitored using a strap-on extensometer with a length of 50 mm. At least six specimens were tested to report the average value, and the samples broken within the grips were not taken into consideration for the strength characterization. In order to estimate the fiber strength, a simple micromechanics model (i.e. rule of the mixture) was used to back-calculate the fiber strength (σ_f) according to Eq. (2).

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

where the subscripts *c, m, f* denote composite, matrix, and fiber, respectively. Because the epoxy used in the study had a higher failure strain than fibers, the conventional matrix strength is replaced by the product of the matrix's Young's modulus (E_m) and the failure strain of composite (ε_c), i.e., $\sigma^*_m = E_m\, \varepsilon_c$. The properties of the epoxy were taken from the previous study (Pisupati et al., 2019). The fiber volume fraction was obtained by following the method in the same reference (Pisupati et al., 2019). Although other micromechanical models considering the shape factor of natural fibers are available (Summerscales et al., 2019), given the large sample size and virtually no difference of shape among the fiber varieties, the simple rule of mixture was used in this study. Statistical analysis was carried out using the analysis of variance (ANOVA) toolbox in Sfipy, Python.

3. Results and discussion

3.1. Yield of fibers

It has been reported in the literature that the yield of flax fibers can be greatly influenced by the amount of nutrients in the soil and the soil treatment (Kumar et al., 2019). For a soil with an average mineralization, it is a rule to apply 70 units of nitrogen minus the amount which is still present in the layer of 0–60 cm in the soil. An excess of nitrogen increases the risk of lodging and the susceptibility to diseases. It delays also the maturation.

In this study, both fields have received a few additional nutrients based on the soil analyses to ensure a good yield. The yield can be related exclusively with the field and the weather without any consideration of additional variables. In Fig. 2, the straw yield of ten different flax varieties for the two consecutive years is presented. It can be clearly noticed that the yield of flax straw in 2018 was approximately twice that in 2017. In general, the rate of germination and emergence of flax crop influences the yield of the fiber (Baley et al., 2019; Goudenhooft et al., 2019a; Karimzadeh Soureshjani et al., 2019). It was identified that an accumulated temperature (θ_{acc}) of about 50 °C is necessary for the germination of flax seeds (Baley et al., 2019). The optimum temperature range for a good germination rate and emergence of flax was reported to be between 20 °C and 28 °C (Karimzadeh Soureshjani et al., 2019). In

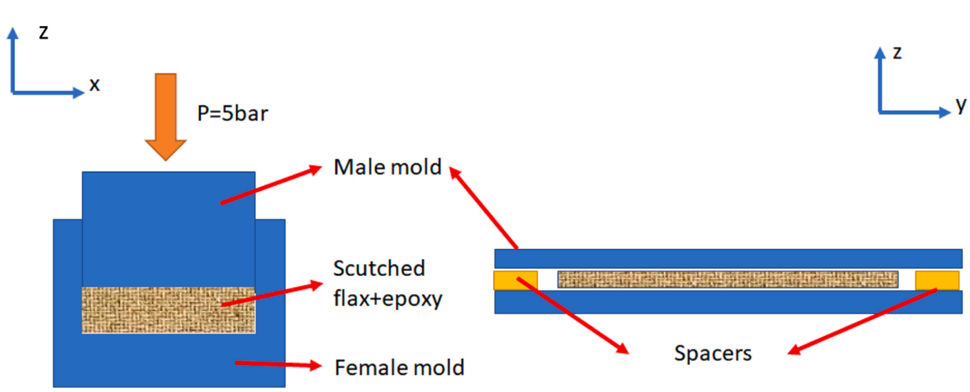

Fig. 1. Schematic of compression molding for manufacturing quasi-UD scutched flax composites: (left) Cross-sectional view along the length of the mold (right) Cross-sectional view along the width of the mold.

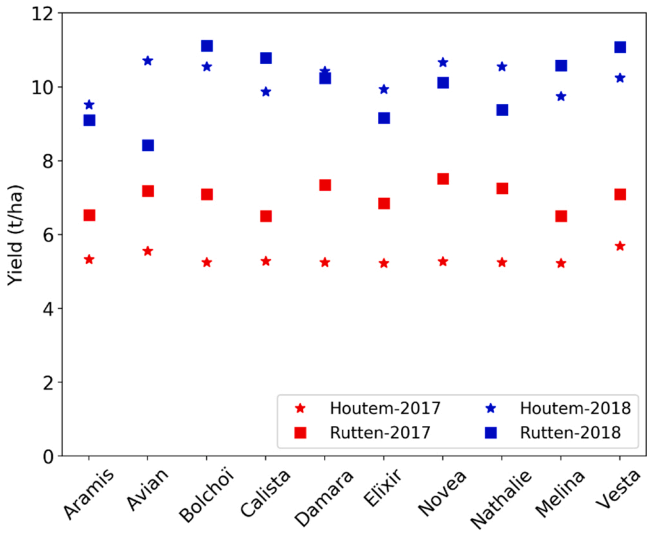

Fig. 2. Yield of flax straw for two consecutive cultivation years i.e., 2017 and 2018.

2017, the emergence of flax was relatively late compared with that in 2018. The time to reach 50 °C of θ_{acc} was between 12 days and 13 days in 2017. In 2018, however, it was between 5 days and 6 days. In fact, the flax was sown three to fours weeks later in 2018 than in 2017. Hence, it was warmer at the moment of sowing in 2018 than in 2017. This difference in θ_{acc} could have greatly influenced the emergence of flax. Furthermore, the temperatures during the emergence period were relatively lower than the optimum temperatures which has lead to a low germination and emergence rate. The flax straw yields in 2017 was similar to the values reported by Bourmaud et al. (Bourmaud et al., 2015).

The statistical significance between both fields was investigated using the ANOVA method (Pugachev, 1984). If the *p-value* is lower than 0.05, there is a significant difference and vice versa. The *p-value* for fiber yield between Houtem and Rutten in 2017 was 1.25×10^{-10} indicating a significant difference of yield between the regions. In 2018, the *p-value* was 0.508, indicating that there was no significant difference of yield between the regions. In 2017, at both trial locations (Houtem and Rutten), the emergence of flax took a relatively long time, whereas the emergence was homogenous for all varieties. This led to the non-uniform distribution of the flax length. In 2017, the mean total length of the flax for ten varieties was 66.5 cm in both the trials. In 2018, the mean total length of the flax for ten varieties was 103.8 cm in Houtem and 95.1 cm in Rutten, respectively. In 2018, the flax showed almost simultaneous emergence at both trial locations. During the early stage of the growing season (i.e. the first two months after sowing) in 2018, the weather condition such as precipitation was relatively favorable (see Table 3).

At the trial location in Rutten, the rapid growth of flax led to an early lodging. Lodging is a physical phenomenon where the flax stem bends towards the ground caused by severe weather conditions or poor soil conditions. In the case of early lodging, there are chances for the plant to overcome this phenomenon. The recovery from lodging during a mature state is very rare however (Goudenhooft et al., 2019b). Recovery from the early lodging was later observed, whereas some flax plants had a bent until the harvest. Flax plants were not lodged in the trial in Houtem. The influence of accumulated temperature on the yield of flax fibers is unclear. In 2017, the accumulated temperatures were 855 °C and 1021 °C in Houtem and Rutten trials respectively, where the yield of these two fields was significantly different (See Fig. 2). On the other hand, in 2018, this difference in the fiber yield has vanished even though the difference of the accumulated temperature between Houtem (907 °C) and Rutten (1138 °C) trials was high (See Fig. 2). In terms of precipitation at the growing season (i.e. from sowing to pulling), Houtem trials experienced rainfall in both years, 58.8 mm in 2017 and 73.4 mm in 2018 (see Table 2). Rutten trials experienced rainfall, 142.2 mm in 2017 and 145.2 mm in 2018. The difference in the yield between 2017 and 2018 can be explained by the relative difference in the monthly rainfall at the eary phase of growth, viz. the first two months after sowing (See Table 3 and Fig. 2).

In Fig. 3, the yield of long fibers, i.e. the ratio of long fibers mass to the total mass of flax straw, obtained from both trials is presented. Given that these fibers are to serve as a reinforcement for high-performance composite materials, the by-products such as flax shives are not considered to evaluate the yield of long fibers. It can be noticed that the long fiber yield was higher in 2018 than in 2017, which is coherent with the straw yield presented in Fig. 2 (See Fig. 3). In 2017, both the fields produced about 13.2 % of long fibers on average, whereas in 2018, the long fiber yield was about 24.9 % on average. It should be noted that the long fiber yield had no significant difference between the fields even though there were significant differences in straw yield between the fields in 2017 and in 2018. The *p-value* for long fiber yield in both fields in 2017 is 0.8026 and 0.5618 in 2018, exhibiting no significant difference.

It is well known that the scutchability of fibers is highly dependent on the retting degree of fibers. If the flax straw is not retted sufficiently during scutching, the fibers can get damaged, and the yield and the mechanical properties can be significantly reduced (Baley, 2002; Bourmaud et al., 2015). On the other hand, the harvest period is also a factor affecting the yield of fibers. In the case of the early harvest, the amount of flax straw obtained will be low (Robinson, 1931) therefore influencing the long fiber yield. As previously mentioned, the long fiber yield is an important criterion in optimizing the retting duration. In Table 4, the dates of pulling and the retting durations for the trials in Beitem in 2017 and 2018 are presented. The fibers were classified based on the harvest period and the retting degree (see Table 4). Three different harvest periods were considered in this study, namely early harvest (E), normal harvest (N), and late harvest (L). The classification number (i.e., 1, 2 and 3 in the fiber index) followed by the harvest period indicated in Table 4 and Fig. 4 refer to the retting degree, viz. 1 for just scutchable, 2 for good retted and 3 for highly retted. In Fig. 4, the long fiber yield in both years is presented. Similar to the yield reported in the previous section (see Figs. 2 and 3), the long fiber yield in 2017 was much lower than that in 2018. It should be noted that in Fig. 4, the fibers are classified by two parameters, namely harvest date (E, N and L) and retting duration (1, 2 and 3. In Fig. 4, the long fiber yield for the early harvest in 2017 trial showed a continuous decrease with an increase in retting duration. This trend can be observed for the other two harvest groups (i.

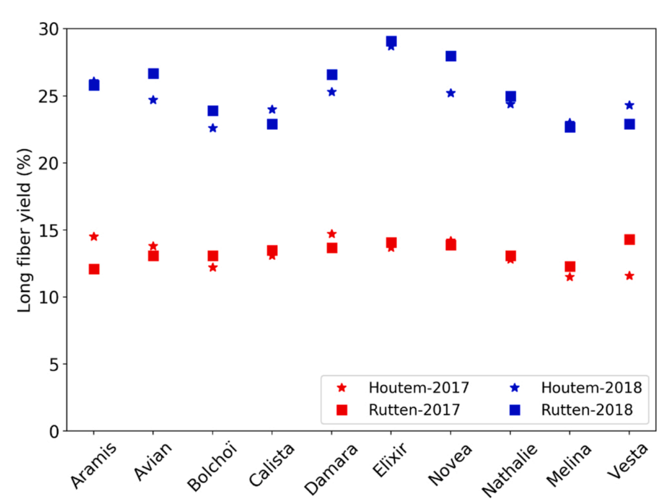

Fig. 3. Long fiber yield for different flax varieties.

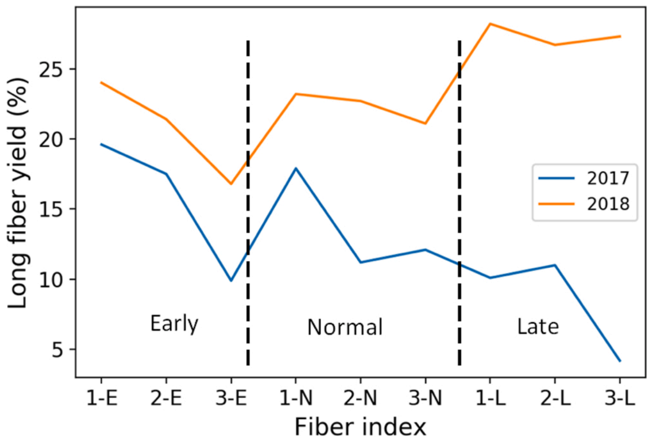

Fig. 4. Long fiber yield for different harvest dates and different retting degrees.

e., 1-N to 3-N and 1-L to 3-L). This decrease in yield can be explained by the absence of stem residues and impurities within the scutched fibers. Because the 1-E fibers were insufficiently retted, it was difficult to separate the fibers from the straw, whose mass was added to the mass of fibers. The similar behavior was also observed for the yield in 2018. The long fiber yield of 3-E fibers was lower than that of 1-E because the over retting of fibers ensured easier straw separation. The lower yield of over retting case can also be explained by the loss of fiber components due to retting. Moreover, the fibers were partially broken off during the retting, in the case of over retting. Those fibers were sucked off to the short fibers during the scutching and were not considered as long fibers. Hence, the over retting resulted in the loss of long fibers.

Given the large difference, however, it cannot be the only contributing factor to a loss in yield (Bourmaud et al., 2018; Liu et al., 2015). Similar to the observations made in the previous section, the yield after retting in 2018 was better than that in 2017 owing to good weather conditions. It can be concluded that the early pulling and lower retting degree can be detrimental to the quality of long fibers due to the presence of impurities in the fibers and also can degrade the mechanical strength of fibers by damaging them during separation. On the other hand, the late pulling contributes no more than the normal pulling as no further development of fibers takes place.

3.2. Measurement of fiber diameter

In Fig. 5, the distribution of fiber diameters for ten flax varieties is presented. The distribution over the two years is similar for each fiber variety, whose variation is similar to the values observed in the literature (Baley et al., 2019; Pillin et al., 2011). The average values lie in the range of 15 ~ 21 μm, with a corresponding *p-value* > 0.05 for all the cases proving that there is no statistical difference for the different cultivation years and flax varieties. On the other hand, for different retting degrees and harvest dates, a difference in the fiber diameter can be observed (see Fig. 6). Firstly, from the early harvest to the normal and late harvest, the average fiber diameter is slightly increased, which is associated with the development of fibers within the plant. Furthermore, the distribution of fiber diameters also depends on the harvest day. In the case of early harvest, the fiber diameter shows a multimodal distribution, whereas a normal distribution can be observed for the normal and late harvest cases. It should be noted, however that the late harvest case has a distribution which has heavy tails (i.e., large kurtosis).(see Fig. 6). These differences in the fiber diameters can greatly influence the tensile strength of flax fibers, which is addressed in the following sections.

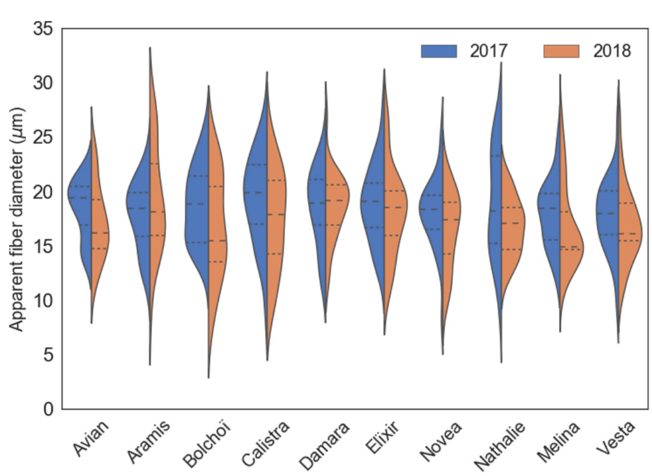

Fig. 5. Apparent fiber diameter distribution of different flax varieties over two consecutive years.

3.3. Tensile test of fiber bundles

Fig. 7 shows the mean tensile strength of flax fiber bundles for the ten varieties over a period of two years at two cultivation fields. The tensile strength of fiber bundles ranges from 20.03 cN/tex to 39.37 cN/tex. The mean tensile strength of all fiber bundles over the two years was 28.69 cN/tex. Similar to the results of yield, the fiber bundle strength seems to be dependent on the growth of flax fibers. For year 2017, the fibers obtained in Houtem exhibited lower bundle strength than in Ruttem except in the cases of Novea and Natalie, even if the difference lies within the standard deviation. In 2018, however, the fibers exhibited an inverse tendency where Houtem trials resulted in better bundle strengths than Rutten trials, except for Vesta and Calista which exhibit negligible difference. This difference may be related to the significant lodging of fibers during the Rutten trials (Goudenhooft et al., 2019b). Among all the ten varieties, Calista showed consistent values irrespective of cultivation year and field.

Statistical analysis using ANOVA was carried out to understand the differences of bundle strength. Considering all four trials (i.e., two cultivation years per two fields) for each variety as test data, the *p-value* was found to be 0.0146 which was below 0.05 indicating that the fiber bundle strengths are significantly different. Furthermore, when the field trials are individually analyzed for each field, the Rutten field trials showed no significant difference between 2017 and 2018 (*p-value* = 0.0659) whereas the Houtem field trials showed a significant difference (*p-value* = 0.0476). This difference between the trials in terms of cultivation year and field can be closely related to the weather condition where the fibers had enough time to develop cell walls and their components, since the other factors such as testing conditions were maintained to be the same. In 2017, the weather condition such as the precipitation at the early stage of growth was unfavorable for flax cultivation at both the trial locations, and the yield was low as observed in Figs. 2 and 3, which also influenced the bundle strength. The flax plants had stunted growth in 2017. For the variation between the two fields in 2017, the *p-value* was found to be 0.541, indicating no significant difference. On the other hand, the trials in 2018 showed a great difference between the two fields (*p-value* = 0.0008) owing to different weather condition such as the precipitation at the early stage of growth (see Table 3). The same tests were performed on fibers with different retting degrees. Owing to the high stem impurities and cortical tissues, the results were not reproducible. Hence, they are not reported in this work.

3.4. Tensile test of composites

In the previous section, the dry fiber bundle strengths of different varieties were presented. From those results, a difference between the varieties could be noticed clearly. The contribution of fiber bundles to

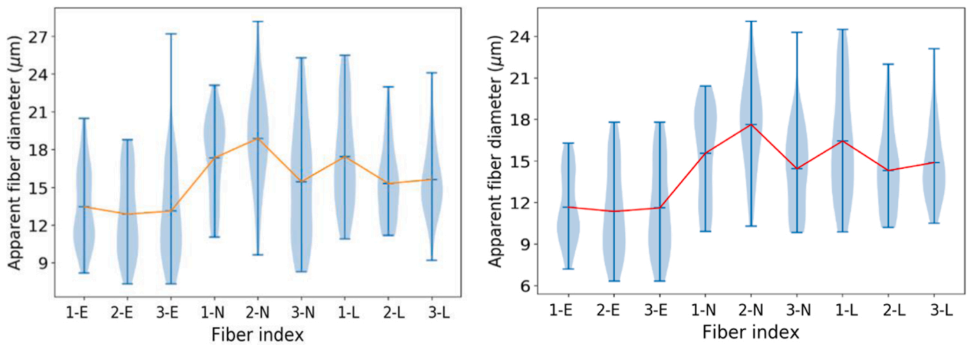

Fig. 6. Apparent fiber diameters for different harvest dates (E- early, N-normal, L-Late) and retting degrees (1-Under retted, 2- Normally retted, 3-Over retted): 2017 (left) and 2018 (right).

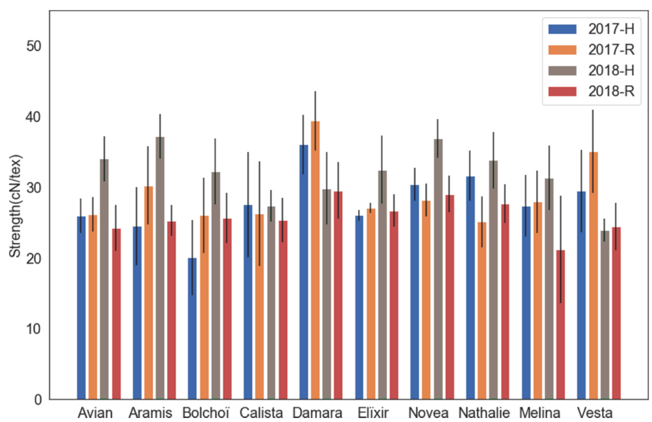

Fig. 7. Fiber bundle strengths of different flax varieties from Houtem (H) and Rutten (R) trails.

the mechanical properties of composites cannot be interpreted solely by the results of dry fiber bundle tests due to several different failure mechanisms other than the fiber failure within a composite. Thus, the tensile strength of composite specimens manufactured with different flax varieties and for different retting degrees was characterized.

In Fig. 8, the tensile strength of composites from different flax varieties can be shown, which is similar to that of the bundle tests. It can be noticed that the Damara variety exhibited higher average composite strength than the others, except for Rutten trials in 2018, and the Novea showed almost same composite strength irrespective of cultivation year

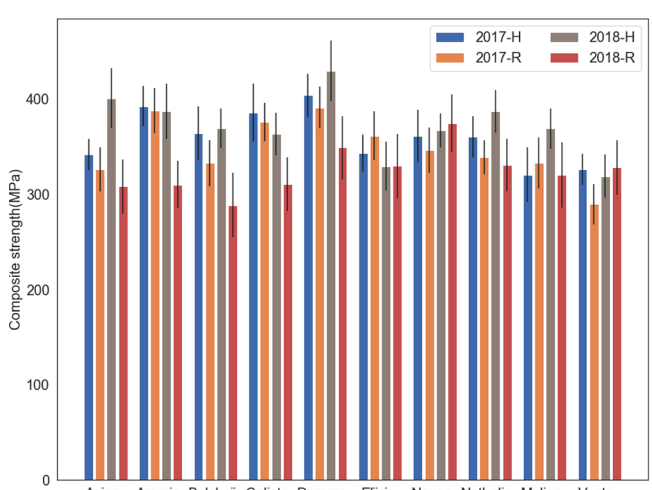

Fig. 8. UD composite strengths from Houtem (H) and Rutten (R) trails.

and region (see Fig. 8). Overall, the trials in Rutten in 2018 provided more uniform values of average composite strength for different varieties than in Houtem in 2018 (see Fig. 8). The difference of flax varieties in terms of composite strength was also observed in the literature (Haag et al., 2017).

Given that it is not easy to draw conclusions about the influence of variety on the composite strength, their statistical significance was analyzed using ANOVA. The resulting *p-values* are presented in Figs. 9 and 10 for both trial fields. For the sake of easier understanding of differences amongst the varieties, the *p-values* were divided into three categories and were color-coded. Green indicates a significant difference between the varieties, i.e. *p-value* $<$ 0.005, blue indicates that there exists a difference between the varieties, i.e. 0.005 $<$ *p-value* $<$ 0.05, and red indicates that the varieties are statistically equivalent, i.e *p-value* $>$ 0.05. For Rutten trials in 2017, many varieties had different tensile strengths and only a handful of them were statistically different to one another (see Fig. 9).

Tensile strength of composites manufactured with flax fibers was improved as the retting degree was increased (See Fig. 11). This trend was also reported by Chabbert et al. (Chabbert et al., 2020). In the case of 1-E composites (i.e., early harvest and insufficient retting), the fibers had a high amount of impurities in the form of stem residues and cortical tissues. This led to a significant decrease in tensile strength of composites owing to poor fiber-matrix interface and lower degree of fiber individualization (Lefeuvre et al., 2015a). This hypothesis can be further supported by the tensile strengths observed for 1-N and 1-L samples (i.e. for the same retting degree, but a later harvest). Furthermore, in the case of 1-E fibers, the fibers were not completely developed, which can be another reason for the low strength. Similar results were observed in the case of hemp fibers in the literature, where the fiber strength was increased according to the increase of retting degree (Liu et al., 2015). On the other hand, the retting degree also had a significant influence on the composite properties. With an increase in the retting duration, the composite properties increased. In the case of late harvest in 2017, the composites from the flax fibers which were over retted as well as those poor retted, exhibited low strength, whereas the intermediate retting degree resulted in relatively good tensile strength (See Fig. 11).

3.5. Estimation of fiber properties

Using the micromechanics model (Eq. (2)), the tensile strength of flax fibers was estimated (see Fig. 12). Given that the porosity content is very small (i.e. about 2%), the rule of mixture for the backcalculation of fiber strength is valid. The estimated fiber strengths (presented in Fig. 12) were significantly higher than the values reported in the literature (Bensadoun et al., 2017). In a recent work, however, similar values for average fiber strength as obtained in this work were observed (De Prez et al., 2019). Overall, the average estimated fiber strength from all the trials is 688.0 MPa. In both trial years, the fibers cultivated in

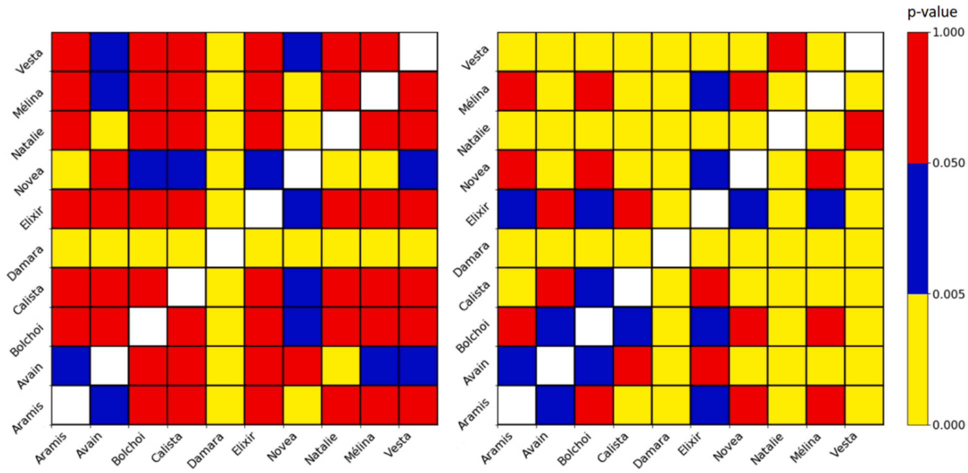

Fig. 9. Statistical significance of the composite strengths in Rutten trials: 2017 (left) and 2018 (right). Green indicates *p-value* < 0.005, blue indicates *p-value* < 0.05 and red indicates *p-value* > 0.05.

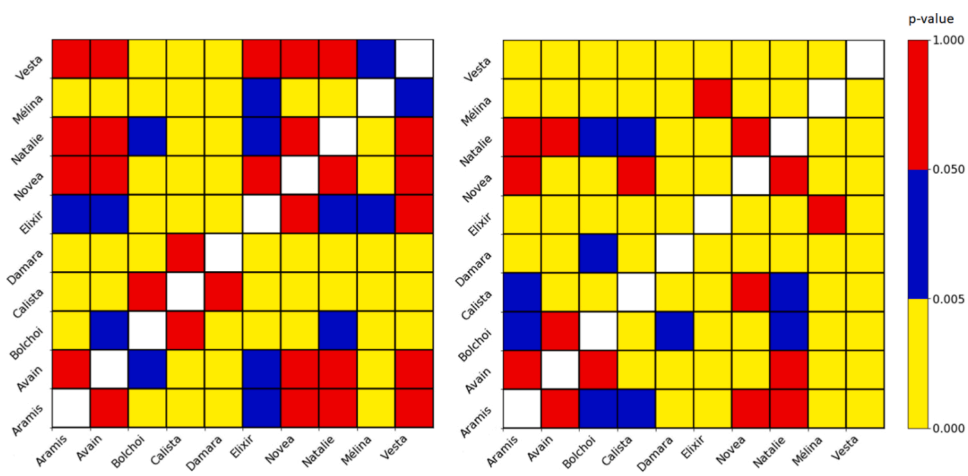

Fig. 10. Statistical significance of the composite strengths in Houtem trials: 2017 (left) and 2018 (right). Green indicates *p-value* < 0.005, blue indicates *p-value* < 0.05 and red indicates *p-value* > 0.05.

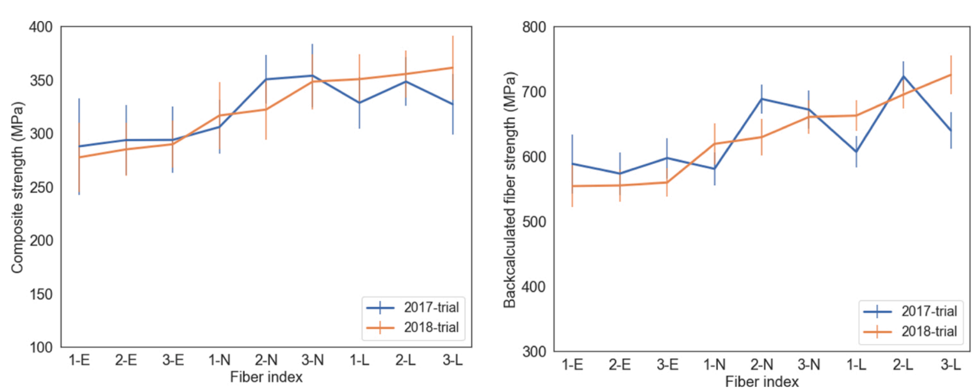

Fig. 11. Composite strength of fibers with different harvest dates [E- early, N-normal, L-Late] and retting degrees [1-Under retted, 2- Normally retted, 3-Over retted] (left). Back calculated fiber strength of fibers with different harvest dates and retting degrees (right).

Houtem had higher average fiber strengths, 716.2 MPa and 726.3 MPa, in 2017 and in 2018 respectively, whereas the average fiber strengths in Rutten trials were 655.9 MPa and 650.0 MPa, in 2017 and in 2018 respectively. Upon a closer look, a significant difference among flax varieties can be noticed. It may be argued that these differences arise from the variation in the composite characterization procedure or the manufacturing process (Bensadoun et al., 2017). Given that the tests were repeated at least six times, the difference in estimated fiber strength values can be attributed to the different nature of in flax varieties. Conversely, with respect to different retting degrees and harvest dates, the estimation of fiber strength by IFBT is not an appropriate method owing to the condition of fibers. Ideally, for the back calculation

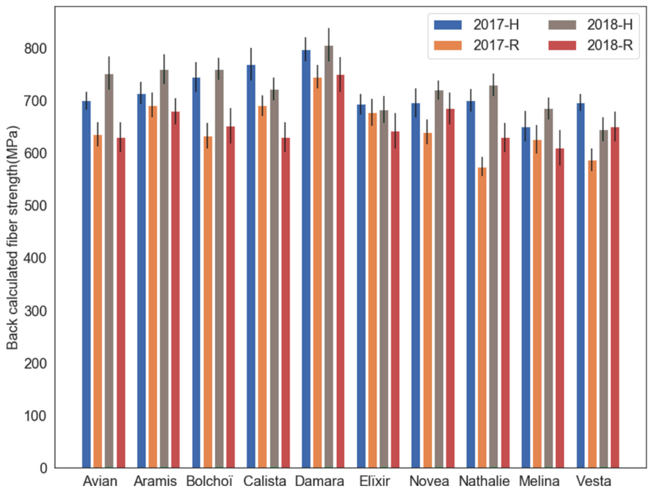

Fig. 12. Back calculated fiber strengths from UD composite strengths for different flax varieties.

of fiber strength, composites should meet the following requirements; perfect fiber-matrix bonding and only two phases (i.e. fiber and matrix). In the case of under retted fibers, the presence of stem residues and cortical tissues makes it difficult to estimate the fiber strength because they may lead to an early failure. Even if three-phase composite micromechanics models are used, it is difficult to characterize the properties of flax shives and their content within the composites. Thus, the estimates are very far from the values observed in the literature (Martin et al., 2013).

4. Conclusions

In this study, the influence of growing conditions and the varietal differences among flax fibers has been presented. From the quantitative analysis of the mechanical property and the yield of flax fibers, it was noticed that the difference of the geographic location has no significant influence on the yield of flax fibers considering that the good soil conditions were ensured for flax growth. On the other hand, the accumulated temperature during the flax germination and emergence can have great influence of the yield of flax fiber and the rainfall at the early phase of flax growth can highly affect the fiber strength and yield. The difference in the overall accumulated temperatures showed little to no influence whereas the accumulated temperature for germination and the period of precipitation affected the yield and tensile strength of flax fibers respectively. A statistical difference in dry bundle strengths between two consecutive years was caused by the difference of growing conditions such as accumulated temperature and precipitatio whereas the same varieties were cultivated in the same regions. Similar results were also observed in the case of composites. Although a couple of varieties of flax fibers can be identified as suitable candidates for composite applications owing to their good properties, it is not always possible to cultivate a single variety. Instead, a mixture of best performing varieties can be a good alternative to serve as the reinforcements in the composites. The suitable varieties can be easily identified using IFBT given that, unlike single fiber tests, the approach of IFBT is more flexible and relatively easier because several thousands of fibers are tested together. This approach is well suited for the screening of fibers for composite application. In this work, the flax varieties cultivated in the two regions in Belgium have been investigated. As a matter of fact, most of the fiber flax worldwide, up to 80 %, is cultivated in the regions of North of France, Belgium and the Netherlands. In those three countries, the same flax varieties are cultivated. Thus, the results are applicable to 80 % of the world acreage of fiber flax. The general conclusion that the growing conditions are more important than variety differences, are applicable to flax grown in other regions around the world.

CRediT authorship contribution statement

Anurag Pisupati: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Visualization, Writing - original draft. **Lies Willaert:** Investigation, Validation, Resources, Data curation, Writing - review & editing. **Frederik Goethals:** Conceptualization, Methodology, Validation, Investigation, Data curation, Writing - review & editing. **Willem Uyttendaele:** Validation, Investigation, Data curation, Writing - review & editing. **Chung Hae Park:** Conceptualization, Methodology, Writing - review & editing.

Declaration of Competing Interest

The authors report no declarations of interest.

Acknowledgments

The authors would like to thank the European Union (European Regional Development Fund (FEDER) for the financial support to the BIOCOMPAL project within the framework of the INTERREG/FWVL program.

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