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Article

Development of 100% Linseed Flax Yarns with Improved Mechanical Properties and Durability for Geotextiles Applications

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Abstract: Due to the ever-growing demand for bast fibres for technical and garment textiles, complementary sources to textile flax, whose cultivation in western Europe cannot really be extended, need to be proposed. In this study, the interest in harvesting and processing linseed flax straw is studied for geotextile applications. The main critical stages of fibre-to-yarn production for geotextiles were investigated. Different dew retting levels as well as different all-fibre extraction processes were investigated to achieve this objective. It was demonstrated that the fibres extracted from linseed flax stems subjected to 12 weeks of dew retting using breaking rollers, thresher and a breaking card exhibited the most suitable morphological and mechanical properties. The optimal fibres were converted into 100% linseed flax yarns using a flyer spinning machine, and the mechanical properties as well as the biodegradability of the linseed yarns were evaluated to understand their potential as geotextiles. These linseed flax yarns were further coated with linseed oil or chitosan to enhance their durability. It was observed that the linseed oil coating better preserved the yarn's integrity and mechanical properties over time, and it permitted doubling their service life potential.

Keywords: vegetal fibre; dew retting; fibre extraction; mean fibre length; coating; bio-degradation

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1. Introduction

In the last few decades, a growing interest has been observed in the use of renewable, bio-based, environment-friendly materials over non-renewable ones. Vegetal fibres are an example of some renewable materials that are widely used as raw materials for manufacturing technical textiles, garments, polymer-reinforced composites, etc. Vegetal fibres are of different types based on their origin. However, at the present time, the major portion of vegetal fibres are collected from the stems of different plant sources and are known as bast fibre. To facilitate the separation of the bast fibres from the woody part of the stem, a biological process called retting (or degumming) is usually performed [1]. The retting of the fibre-rich stems can be conducted in five different ways: dew retting, water retting, enzymatic retting, chemical retting and mechanical retting. Among these retting processes, dew retting is mainly performed in western Europe. In case of dew retting, straws are cut or pulled before being laid on the fields for durations ranging from a few days to a few weeks, depending on the type of bast fibres and the expected level of retting. Weather conditions (temperature, morning dew, rainfall, humidity, soil composition, soil pH, retting time, etc.) are the factors that control the dew retting process [2–4]. Dew retting alters the physicochemical and mechanical properties of the bast fibres; thus, it needs to be conducted in a controlled manner [5]. There is a significant amount of information available about the retting of textile flax and the properties of textile flax fibres, but the information about linseed flax fibres is rather limited [6].

Linseed flax is primarily cultivated for its seeds. After seed harvesting, the linseed flax straws, which are rich in good quality fibre, are generally buried in the fields or burnt. This does not constitute a good practice for the environment and from an economic point of view [7,8]. To facilitate seed harvesting and prevent lodging, a chemical shortener during the growth of linseed flax is often used. Hence, the straw yield available for the production of fibres is generally low (0.4 t/ha), and this straw is generally poor in fibres, as a large part of it is constituted by fibreless branches that support the seeds [9]. Nonetheless, linseed flax straw may still be a large source of fibre if the shortener is not used or in organic farming [10]. Diederichsen and Ulrich [11] reported that the fibre content of linseed flax straws is around 27%, while in the case of textile flax (in their study), it is around 34%. Ouagne et al. [12] reported that the fibre content in linseed stems (not submitted to chemical shortening) varies between 37% and 40%, which is greater than all other results reported in earlier literature [10].

The straw length of the linseed flax is comparatively small (between 40 cm and 70 cm), and they are left randomly aligned on the ground following the seed harvest by a combine harvesting machine. Hence, the extraction of fibre from linseed stems through traditional scutching and hackling devices is inappropriate as these processes require long and aligned straws. Xu et al. [13] used a hammer mill device to extract fibre from linseed stems. This device effectively extracted the fibre from the linseed stems, but its aggressive action deteriorates the mechanical properties by means of incorporating kink-band defects into the extracted fibres [14]. Ouagne et al. [12] extracted fibres from linseed flax straw using an “all fibre” extraction device where the extracted technical fibre length varied between 3.7 and 5.3 cm. The mechanical properties of these extracted fibres were comparatively low (tensile strength of 377 MPa) than that of the manually extracted linseed fibres of the same variety (1086 MPa) [15]. Ouagne et al. [12] concluded that the dew retting of the linseed flax needs to be optimised prior to fibre extraction. Recently, Khan et al. [16] extracted fibres from six-week-rated linseed flax straws via a method that consists of breaking by compression and threshing straws followed by breaker carding. They observed that the new fibre extraction process is much better at preserving the length and mechanical properties of the extracted linseed fibre bundles in comparison with the previously proposed linseed flax fibre extraction devices [12,17]. However, according to Khan et al. [16], a retting time of only six weeks is still relatively short, and they suggested that much longer dew retting durations (at least three months) should be applied for future work in their case study.

Although linseed flax fibres may have huge potential in different areas, such as load-bearing composite materials [17], their present consumption is mainly limited to insulation panel manufacturing, non-load-bearing composite reinforcement and paper industries. In these aforementioned applications, linseed flax is used in fibre form. However, if these fibres were converted into technical yarn, they could find huge potential in different areas, including geotextiles. Although, at present, synthetic fibres are dominating the geotextile market, the presence of natural fibres in geotextile applications cannot be denied. The main advantages of natural-fibre-based geotextiles are their good mechanical properties, low cost, and renewable and environment-friendly characteristics. The fibre requirements in geotextiles depend mainly on their applications. The use of natural fibres in geotextiles is mostly for short-term reinforcement and erosion control applications, as these geotextiles should be decomposed once the soil is stabilised by plant roots [18]. Coir is the main natural fibre used for geotextile applications in Europe, but the vast majority of coir nets is imported from Asia. In the future, it would be judicious to be able to produce geotextiles for the European market with local resources, which would improve the carbon footprint of the manufactured product by reducing the distance it would have to travel between its place of production and place of use. Initial works on oleaginous flax fibre (from the southwest of France) showed that it could be used for the manufacture of yarns and geotextiles [12,16]. Nevertheless, the mechanical properties of the individual fibres, as presented in the existing literature, suggest that improvements are still possible, in particular, by applying longer field retting times to the straw before its collection [12,16]. Moreover, it will be necessary in the future to adapt the life span of geotextiles in real conditions to the needs expected by the applicator. Indeed, Lekha [19] placed a coir net in the soil to test its biodegradability, and it was concluded that it lost 68% of its tensile strength in seven months. In parallel, some treatments applied to fibres prior to their use in geotextiles could increase their life in the soil by inhibiting the activity of cellulolytic soil bacteria and/or by increasing their hydrophobic character. Low-molecular-weight chitosan is able to protect textile flax-fibre-based yarns against natural soil biodegradation when impregnated on the fibres, preventing the degradation of amorphous and crystalline cellulose, hemicelluloses and pectins [20,21]. As a reminder, chitosan is a polyoside produced from chitin, the component of the exoskeleton of crustaceans. Page et al. [22] also evidenced that the treatment of textile flax fibres with linseed oil for fibre-reinforced cementitious composites reduced their water absorption. Although the materials described in this recent study were not geotextiles, such a solution would be worth considering for the future of geotextile products based on linseed flax fibres before they are placed in soils.

To the best of our knowledge, no scientific study has used linseed flax fibres for the manufacture of natural geotextiles. This work will therefore be the first contribution to obtaining linseed flax yarns intended for the subsequent manufacture of geotextiles. More precisely, this study will aim to optimize all the parameters, from the field to the final product, to produce yarns based on 100% linseed flax fibres for subsequent geotextile applications. These parameters include the combine harvester settings at the moment of the straw harvesting, the dew retting duration of straw (up to 14 weeks) before its collection in the field, the mechanical extraction methodology applied for continuously extracting fibre bundles from straw, the yarn manufacturing using a flyer spinning frame and the effect of coating treatments (with chitosan or linseed oil) on the evolution of the yarn's tensile properties over time when they are positioned in the soil to simulate the actual conditions of use of a geotextile.

2. Materials and Methods

2.1. Raw Materials

For this present study, the straws of oleaginous flax (*Linum usitatissimum* L.) were provided by the Ovalie Innovation Cooperative, Auch, France. This oleaginous flax was cultivated in summer 2019, in the southwestern part of France (Gers department), and it was from the Angora variety. More precisely, the four batches of straw tested in this study came from the same plot, located in Saramon (43°52′ N, 0°77′ E). A combine harvester was used for the seed harvesting, which took place on July 13, 2019. The straws were sprayed in the field for dew retting at the same time. The four straw batches corresponded to two different settings for the combine harvester, for which the shredder was disconnected to preserve both the length of the stems and the mechanical strength of the bast fibres. On the one hand, a high (i.e., a normal) cut (i.e., 30 cm above the ground) was chosen for batches 1 and 3, corresponding to a mean length for stems of around 35 cm. On the other hand, a low cut (i.e., only 5 cm above the ground) was chosen for batches 2 and 4, corresponding to a mean length for stems of around 60 cm. In parallel, two different dew retting durations were tested, 12 weeks for batches 1 and 2 (straw collection on 30 September 2019) and 14 weeks for batches 3 and 4 (straw collection on 16 October 2019). These retting durations were deliberately chosen to be much longer than in a previous study also conducted on oleaginous flax straw (6 weeks max.) [16]. For each batch, straw harvesting was conducted in three adjacent areas of the plot, each 50 m long and 5.6 m wide, separated from each other by a 5 m gap. This methodology allowed for the calculation of straw yield (in ton/ha) three times. The results for straw yield were presented as mean values ± standard deviations. Harvesting details of the four batches of oleaginous flax straw used in this study are given in Table 1.

Table 1. Harvesting details of the four batches of oleaginous flax straw used in this study.

Batch Number	Cutting Type	Dew Retting Duration (Weeks)
1	High cut	12
2	Low cut	12
3	High cut	14
4	Low cut	14

2.2. Manual Extraction of Fibres

Around 200 g of oleaginous flax straw was chosen randomly from each batch, and the bast fibres and shives were then manually separated from each other. The mass content of the fibres in each batch of oleaginous flax straw was determined at the end, and the result was expressed as a mass percentage of the straw. The manually extracted fibres were then used to determine their chemical composition.

2.3. Chemical Composition of Fibres

Vegetal fibres contain moisture. The moisture content of the manually extracted fibrous materials was initially determined using the ISO 665:2000 standard [23]. These fibres were used for the analysis of their chemical composition. In this regard, these fibres were cut to a length of 4–5 mm with a pair of scissors. Then, a Foss (Hillerød, Denmark) Tecator Cyclotec 1093 mill grinder was used to grind the fibres into a fine powder using a 1 mm sieve. This fine ground powder was used for the chemical composition characterization.

The ADF-NDF (ADF for acid detergent fibre and NDF for neutral detergent fibre) method developed by Van Soest and Wine was used to determine the three main parietal constituents inside the fibres, i.e., lignins, hemicelluloses and cellulose [24,25]. A Foss Tecator FT122 Fibertec hot extractor was used for the hot extractions, and a Foss Tecator FT121 Fibertec cold extractor was used for the cold ones.

Pectin content was determined using a non-normalized two-step method:

(i.) An alcohol-insoluble solid fraction was first obtained using a Foss Tecator FT122 Fibertec hot extractor after 3 min boiling in ethanol (95 °C), filtration, rinsing with ethanol (80 °C) and then drying.

(ii.) Pectins were extracted, and they were then transformed into galacturonic acid through their acid hydrolysis; for these two simultaneous actions, 30 min of boiling in 100 mL HCl 0.05 M was required, and the extraction was conducted twice.

The colorimetric method of Baley and Asboe-Hansen was used at the end to quantify galacturonic acid in the extract [26]. In fact, pectins are polymers of galacturonic acid, i.e., polygalacturonic acids.

Around 1 g of powdered fibre was used for the determination of lignin, hemicelluloses and cellulose, and around 500 mg of fibre sample was used for measuring pectins. All determinations were conducted twice. Results were expressed as mean values ± standard deviations in proportion to the dry mass of fibres.

2.4. Mechanical Extraction of Fibres

Two different mechanical extraction methodologies are used for the continuous extraction of fibres from the four oleaginous flax straw batches. These methodologies are denoted as method A and method B. A detailed description of these two methods is given in the paragraphs below.

2.4.1. Method A

This method of fibre extraction involves two main steps: first, the breaking or threshing of the linseed stems, followed by their carding. The extraction of fibre using this method was performed at the facilities of the Hemp-Act company (Lacapelle-Marival, France). A schematic diagram of the breaking and carding zone of the device is shown in Figure 1a. In the breaking device (Figure 1b), a few pairs of rollers are placed horizontally, while some others are placed in an inclined manner. Hence, when the linseed stems pass through this breaking device, the wooden core of the stems breaks into pieces, and a major portion of these broken stems (shives) fall down due to the action of gravity. The output of the breaking device is then fed to the next device, the carding device (as shown in Figure 1c). At this level, some additional shives were removed due to the applied carding action. The fibres were simultaneously refined in diameter, and a web was formed (Figure 1d) at the breaking card outlet, which was rolled up in the form of a large count sliver (Figure 1e) and collected for further experiments.

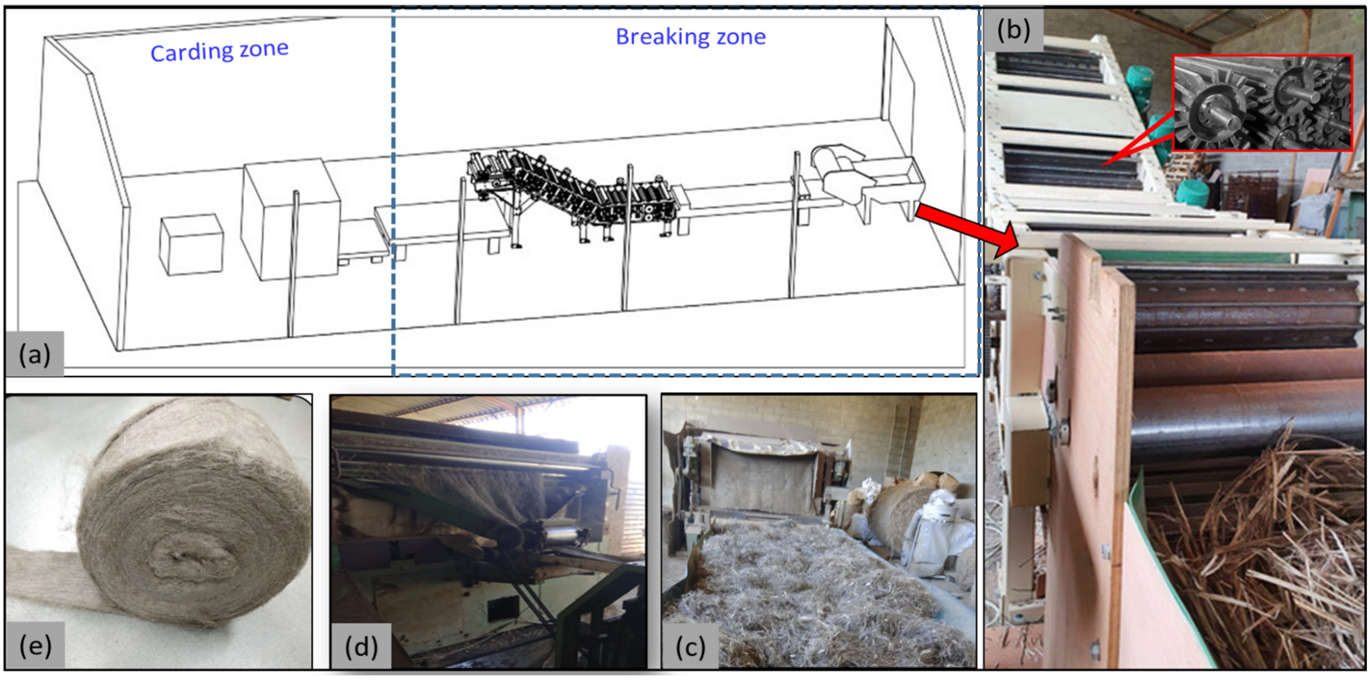

Figure 1. (**a**) General representation of the device used for method A; (**b**) front view of the breaking roller machine; (**c**) fibres at the output of the breaking roller and input of breaking card; (**d**) carded web at the output of the breaking card; (**e**) carded fibre sliver.

2.4.2. Method B

The main equipment for method B was a Laroche Cadette 1000 (Laroche, Cours, France) "all fibre" extraction device (Figure 2). It was used for the mechanical extraction of fibres and then completed an extra sieving step to remove additional shives for better purity in fibres inside the fibrous lap. The 1 m width of this equipment associated with the 3.5 m/min feed belt speed used during the experiments made it possible to process straws at a 175 kg/h inlet flow rate, making this equipment a pilot scale one. The opening and cleaning of the fibres, along with the web formation, are the characteristics of this tearing machine. It consists of three modules with a pair of rollers at the start of each module for smooth feeding of the material. One roller is smooth, and the other grooved roller is made of rubber. The first cylinder of each module has spikes (i.e., nails) to extract fibres from the straw. This cylinder thus consists of a fibre extraction roller, and its speed varies from 750 to 1800 rpm. Trap doors are also situated at the bottom of each module to collect some parts of the shives due to gravity. For the present study, the trap door opening was maximal to ensure as much removal of shives as possible. A perforated cylinder is also situated at the end of each module. These perforated drums have three main functions: (i) the removal of dust due to aspiration, (ii) the web formation, and (iii) the transfer of the fibrous material to the next module or to the machine outlet (case of module 3).

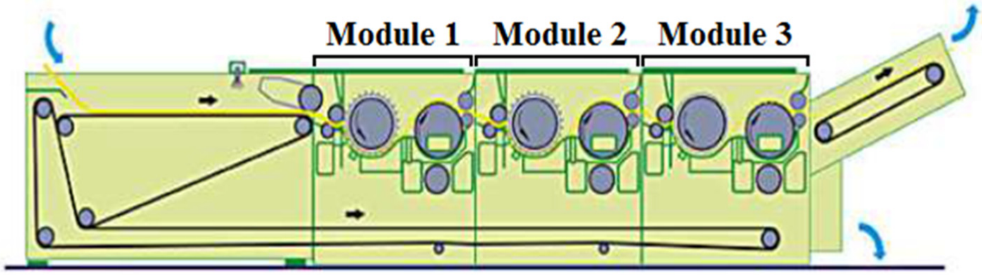

Figure 2. Laroche Cadette 1000 "all fibre" extraction device used in this study (from Laroche company website, https://laroche.fr, accessed on 21 June 2021).

The operating parameters used in this study for modules 1 to 3 (as tabulated in Table 2) were the same as those optimized by Ouagne et al. [12] in a previous study related to the processing of non-retted oleaginous flax straw samples. In addition, a 1.8 m/min speed was chosen for the output belt. After the "all fibre" extraction equipment, a Ritec 600 (Ritec, Signes, France) vibrating sieving machine was used for the extra sieving step. It was fitted with a 12 mm sieve. The objective of this extra step was to eliminate as many shives and as much dust as possible that was still trapped inside the fibrous lap, although some short fibres could also be lost during this step due to their very small size.

Table 2. Operating parameters used for the "all fibre" extraction device (same parameters used as those developed in Khan et al. [16]).

	Module 1	**Module 2**	**Module 3**
Transmission speed of the lap (m/min)	2.2 (from module 1 to module 2)	1.5 (from module 2 to module 3)	
Rotation speed of the extracting roller (rpm)	725	725	725
Rotation speed of the motor for the aspiration at the level of the perforated cylinder (rpm)	1500	2000	2000

2.5. Purity of the Fibrous Materials

After the collection of fibrous materials from the two different mechanical extraction methods described above, the fibres still contained some remaining shives and dust as impurities, so their purity was evaluated. A 100 g test sample mass of the fibrous materials was randomly taken, and shives, dust and fibres were separated manually and weighed. Then, the content of the impurities inside the fibrous lap was calculated as its purity in fibres, which was expressed as a percentage by weight.

2.6. Morphological Analysis of the Mechanically Extracted Fibre Bundles

Morphological analysis of the vegetal fraction obtained at the outlet of each device was performed for each treated batch. Some extracted fibres were randomly taken from different locations. Then, 200 fibre bundles per batch were measured to calculate their lengths. Fibre length was determined by fixing one end of the fibre bundle and linearly extending the other. The actual length was then measured using a double decimetre with an accuracy of 0.5 mm. Then, the average length of the fibre bundles was calculated as well as the associated standard deviation.

2.7. Elementary Fibre's Tensile Testing

A Diastron Ltd. Lex 820 (Diastron Ltd., Andover, UK) automated high-precision extensometer was used to measure the diameter, tensile strength and elastic modulus of elementary (i.e., individual) fibres. To prepare the sample for testing, the individual fibres were manually extracted with care from the bundles using a method involving hot water [27]. Then, they were mounted on dedicated plastic tabs with a length of 12 mm with the help of special glue. To measure the fibre cross-section, the prepared samples were placed on an ombroscopy device that measures projected diameters during a 360° rotation of the fibre. As bast fibres are most of the time not circular, it is now well accepted to model them using an elliptical model, with the fibre cross-section calculated from the small and large mean values of diameters recorded at a minimum of 10 positions along the fibre [14,27].

The sample was then placed in the high-precision extensometer of the testing machine to measure the tensile strength and elastic modulus of the elementary fibre. A 20 N load cell was used for the force measurement applied on the sample with the help of a stepping motor used for traction to break the individual fibre. The speed of the tensile test was 1 mm/min with a displacement accuracy of 1 µm. Recorded points were measured with a 20 ms periodicity. Tensile strength and elastic modulus were measured on forty samples for each batch, allowing, at the end the calculation, for the related mean values and standard deviations. More details about the used devices can be found in [14,27].

2.8. Yarn Production

A Trytex (Coimbatore, India) gill-flyer spinning frame was used to produce yarns from the fibre bundles. A schematic diagram of this device is shown in Figure 3. It is a specially designed laboratory machine with a feed belt, combing rail, drafting zone and special peripheral to cause twisting along the yarn. The control panel of the machine allows the setting of the feed speed and delivery rollers to adjust the sliver to the requirements for obtaining the yarns. One hundred percent pure linseed flax fibres were weighed, and they were then uniformly fed at the input of the gill-flyer spinning machine. A uniform sliver was obtained at its outlet. The doubling and even tripling of the slivers were made to achieve the required sliver counts and, in the end, to obtain the required linear mass value for yarns. By increasing or decreasing the speed of the spindles, one could increase or decrease the twist per meter of the yarns. Around 4 ktex yarns were achieved with a twist level of around 100 tpm (i.e., twists per meter).

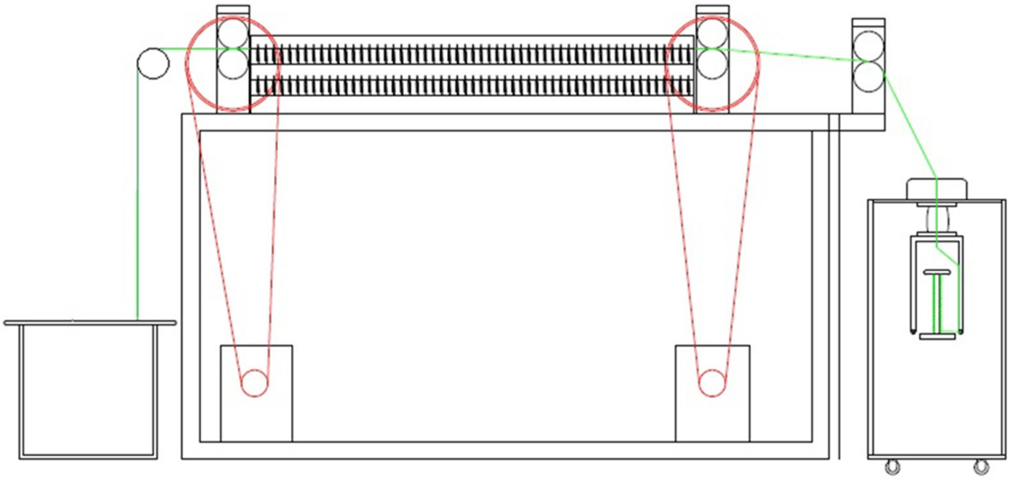

Figure 3. Schematic diagram of the gill-flyer spinning frame.

2.9. Yarn Tensile Properties

The yarns to be tested were cut to a 25 cm length according to the specimen requirements, and they were then fixed on the specimen holder of the machine. A 3 kN load cell was used for the force measurement applied to the sample. During the tensile test, the speed was 1 mm/min until the yarn was broken, deducing its breaking force. Five replications were tested for each of the yarns. More replications would probably have been desirable. In view of the total length of the linseed flax yarn produced, it was, however, not possible to make more than five replications. Even with five replications, the standard deviations obtained for tensile properties were never more than 20% of the associated mean value, which was considered acceptable for a yarn based on natural fibres. The results for breaking force and tensile strength were presented as mean values ± standard deviations.

2.10. Yarn Coating and Durability Tests

To assess the service life of the yarns in real soil conditions, two different coating post-treatments were applied in addition to the non-coated yarns. Yarns manufactured from 100% linseed flax fibres and with linear mass values of 4060 tex were used for these durability tests. They were cut into equal lengths of 25 cm, and all tested yarns had knots at each end. In total, 108 yarn samples were prepared, and 36 of them were used as such, corresponding to the reference yarns (i.e., non-coated yarns). The other yarns were used for the coating treatment, one half with chitosan and the other with linseed oil.

Chitosan from the Kalys company (Bernin, France) was used to prepare a 1% (w/v) chitosan solution in 2% acetic acid. To facilitate the chitosan solubilisation in the acetic acid solution, it was heated up to 75 °C using a magnetic stirrer. Then, the 36 yarn samples were dipped in this solution for 8 h for coating. Coated samples were then placed in a metallic grid, and the grid was positioned in a ventilated oven for 48 h at 50 °C to allow the water to evaporate. The coated samples were then placed in a climatic chamber (25 °C, 65% relative humidity) for 72 h before being placed in the soil for durability testing.

A total of 800 g of linseed oil obtained by first cold pressing of flax seeds was mixed with 200 g of pure gem turpentine oil and a drier at a 15 g/L volume concentration, all three from the Onyx company (Roche-lez-Beaupré, France). Based on cobalt salts and C6 to C19 branched fatty acids, the drier was added to accelerate the drying of linseed oil. For its side, turpentine was added to make the mixture more fluid and to further reduce the drying time. Then, 36 yarn samples were dipped in this solution for 120 s. To remove the excess linseed oil, these samples were placed in a metallic grid at ambient temperature for 24 h. Furthermore, the oil was removed by rolling the samples between paper towels with great care.

Four yarn samples of each type (i.e., non-coated yarns and yarns coated with chitosan or linseed oil) were kept for the yarn's tensile strength measurements. The others (32 per type) were buried in the soil at a depth of about 5 cm in order to simulate the application of a geotextile in real-life conditions. Three separated zones of an equal area were set up, one for each type of yarn sample, and the yarn samples were all aligned with each other and separated by a distance of 10 cm to ensure full contact with the earth. At well-defined times (1, 2, 2.5, 3, 4, 5, 6 and 8 months), four yarn samples of each type were recovered from the soil, and they were then mechanically tested in order to follow the evolution of their tensile strength during the durability tests.

2.11. Yarn Observation through Microscopy

A Keyence (Bois-Colombes, France) VHX-6000 digital microscope was used for observing non-coated and coated yarns before and during the durability tests conducted in the soil. Its camera has a progressive scanning system with a 1/1.8-inch CMOS image sensor. For the yarns taken from the soil during the durability tests, they were first cleaned gently with a brush before being observed under the digital microscope in order to remove any soil particles they may have had on their surface. The size of the images obtained was 20,000 × 20,000 pixels.

3. Results and Discussions

3.1. Contents of Fibres and Shives in the Straw Batches

The linseed flax straw yields as well as the fibre and shive contents of this straw are presented in Table 3. The fibre and shive results were obtained after the manual extraction of fibre from the straw.

Table 3. Contents of fibres and shives in the different straw batches tested (determination through manual extraction).

Batch Number	Straw Length (cm)	Straw Yield (ton/ha)	Fibre Content (mass %)	Shives Content (mass %)
1 (high cut)	35 ± 2	0.97 ± 0.13	30.4 ± 1.8	69.6 ± 2.1
2 (low cut)	59 ± 4	1.82 ± 0.24	32.8 ± 1.3	67.2 ± 1.6
3 (high cut)	34 ± 3	0.98 ± 0.18	28.8 ± 1.7	71.2 ± 2.0
4 (low cut)	61 ± 3	1.88 ± 0.17	33.6 ± 1.2	66.4 ± 1.5

Depending on the position of the cutting bar, the length of the straw is very different and leads to very contrasted straw yields. The straw yield is globally twice as high for the low cut than the high cut. This corresponds to stems that are about twice as long than the ones of the high cut. The fibre content inside the dry mass of straw was in the 29–34% range, and these results were in perfect accordance with some other ones already cited in the literature for linseed flax: 27% [11], 25–30% [10] and up to 29–33% [16]. Although the levels obtained in the present study are lower than in another one, i.e., 37–40% [12], they confirm that oleaginous flax straw can indeed be considered an important source of bast fibres. Comparing the results of this study with each other, it can be further observed that the low cut resulted in straw batches with higher fibre content. These results show that the proportion of fibres along the stem is higher than in the lower part. A low cut also leads to higher straw yields and more important masses of harvested fibres per hectare, with a potential of up to 0.63 kg/ha for batch 4. This potential resource of fibre is lower than the one obtained in the frame of textile flax cultivation with total fibre yields, which are classically situated between 2 and 3 tons/ha [27] as the straw length is lower in the case of linseed flax, and also because the sowing density is lower in the case of linseed flax to leave more space for more voluminous inflorescences. Such a setting of the combine harvester should be preferred for future industrial exploitation although care should be taken as the harvesting machine could be damaged by stones or other hard materials on the ground than with a normal (i.e., higher) cutting height.

3.2. Chemical Composition of Fibres

At the level of the microstructure of linseed flax stems, the cell wall and middle lamella are mainly composed of cellulose, hemicelluloses, lignins and pectins. The chemical composition of the fibres is given in Table 4.

Table 4. Chemical composition of manually extracted fibres as a function of the dew retting duration, including the values from Khan et al. [16] corresponding to no dew retting and six weeks of dew retting (% of dry matter).

Batch Number	Dew Retting Duration (Weeks)	Hemicelluloses	Lignins	Cellulose	Pectins
Ullah Khan et al. [16]	No dew retting (reminder)	12.9 ± 0.5	5.3 ± 0.4	62.1 ± 0.4	4.4 ± 0.2
Ullah Khan et al. [16]	6 (reminder)	10.3 ± 0.0	5.5 ± 0.1	67.0 ± 0.1	3.7 ± 0.2
1	12	8.0 ± 0.4	3.6 ± 0.3	73.9 ± 0.6	3.0 ± 0.3
2	12	8.5 ± 0.4	3.8 ± 0.1	76.5 ± 0.2	2.6 ± 0.1
3	14	6.8 ± 0.7	3.8 ± 0.2	75.7 ± 0.4	2.7 ± 0.3
4	14	3.4 ± 2.2	4.5 ± 1.1	76.8 ± 0.4	2.5 ± 0.1

Results are presented as mean values ± standard deviations.

In Table 4, the chemical composition of manually extracted fibres is presented as a function of the dew retting duration. This table also includes the values from Khan et al. [16] corresponding to no dew retting and dew retting at six weeks. If the straws in this study were from the 2019 harvest, those presented in Khan et al. [16] were cultivated during the summer 2018 but using the same Everest variety and in the same geographic area. When comparing these results, a clear reduction in the pectin content (from 4.4% to 2.5%) was observed with an increased dew retting duration (up to 14 weeks). These results allow us to draw two conclusions. On the one hand, dew retting is a particularly slow process. On the other hand, the choice made for this study more than doubled the duration of dew retting (14 weeks instead of 6 weeks) actually led to the reduction of the pectic cement in higher proportions. A significant reduction in the contents of the hemicelluloses (from 12.9% to 3.4%) and, to a lesser extent and in a less obvious way, of the lignins was also observed during the retting process. In contrast, the cellulose content inside the fibres was logically increased from 62.1% with no dew retting to 76.8% after 14 weeks of dew retting.

It has to be mentioned here that the amount of rainfall affects dew retting effectiveness. The four straw batches in this study were cultivated during summer 2019 when the cumulative rainfall was 150 mm during the 14 weeks of retting, whereas the batches from Khan et al.'s [16] study were from the previous year (2018) when the rainfall was only 78 mm during the 6-week retting process. So, it is reasonable to assume that the duration plays a role in efficient dew retting, and more rainfall seems to allow for more efficient dew retting.

To conclude, dew retting is a particularly important parameter for the subsequent fibre mechanical extraction and further processes. In this study, much higher dew retting durations were applied in comparison with a previous study [16], i.e., 12–14 weeks instead of 6 weeks max., and their effectiveness in reducing the pectin content in fibres was clearly demonstrated. Although the 14-week duration showed slightly better results, the difference in cellulose contents between 12 and 14 weeks of dew retting remained particularly limited. The 12 weeks of dew retting could thus be considered sufficient in this study, allowing the farmer to free his land plot more quickly for tillage and then the following crop.

3.3. Fibre Purity of Extracted Fibre Bundles from Method A

Method A was an arranged as a sequence of horizontal breaker rollers, a thresher and then a breaking card. In this method, the sampling of fibre bundles to measure their purity was not realised after each step but only inside the fibre sliver collected at the outlet of the breaking card. Purity in the fibres of all slivers is given in Table 5, and it varied from 73% to 86%. The minimal purity value (i.e., 73%) was obtained from batch number 3. This straw batch was produced using a high (i.e., normal) cutting height, and it was surprisingly associated with a long dew retting duration of 14 weeks. This low purity value could be the consequence of a more uncontrolled and inhomogeneous retting process, as evidenced by a slightly higher and more dispersed pectin content in the fibres (2.7 ± 0.3%) than for batch number 4 (2.5 ± 0.1%), which was also dew retted for 14 weeks. With a slightly higher pectin content, fibres and shives were more bonded together, which could have therefore led to a lesser elimination of shives during the extraction of the fibres. When comparing the results in this study with those obtained by Khan et al. [16] using the same three-step dry mechanical extraction process, 66–67% fibre purity values were obtained from non-retted straw batches. The results in this study thus clearly evidence the key role played by long dew retting durations in fibre purity.

Table 5. Comparison of the final purities in fibres (percentage) for the two processes tested (i.e., A and B).

Batch Number	**1**	**2**	**3**	**4**
Process A (%)	81.7	81.8	72.6	85.5
Process B (%)	73.4 (−8.3)	73.3 (−8.5)	76.6 (+4.0)	79.7 (−5.8)

The number in parentheses is the difference between the final purity in fibres in process A and B for the same straw batch.

Straw batch number 4 showed the maximum fibre purity, i.e., 86%. This batch was a low-cut one having undergone a 14-week dew retting duration. It was also the batch with the lowest pectin content (i.e., only 2.5%) inside the fibres. It can thus be concluded that a longer dew retting duration promoted a better separation of fibres and shives, thus contributing to a higher fibre purity in the sliver obtained at the breaking card outlet. In parallel, as batch number 4 was harvested using a low cutting height, the straw yield was higher just like the fibre content inside the starting straw (Table 3), thus leading to the production of a larger quantity of extracted fibres with more purity. Finally, although batches number 1 and 2 were retted for only 12 instead of 14 weeks, the fibre purity of extracted fibre bundles was 82% in both cases (Table 5), which is high enough for further processing of the extracted fibres bundles into yarns and then for geotextile applications.

3.4. Fibre Purity of Extracted Fibre Bundles from Method B

Method B was composed of a Laroche Cadette 1000 “all fibre” extraction device for the continuous extraction of fibre bundles and then an extra sieving step, which was useful for removing additional small shive particles still trapped inside the fibrous lap at the Cadette machine outlet. The fibre purity inside the lap at the outlet of the “all fibre” extraction device varied from 50% to 63% (Table 6) as compared with the content in the fibres of the starting straws, which was between 29% and 34% (Table 3). The minimum purity levels after the extraction device (i.e., 50% and 54%) were observed for batches number 1 and 2, respectively, which were both dew-retted for only 12 weeks. Thus, contrary to what was previously observed with the breaking card in method A, it seems that it was more difficult to extract the fibre bundles and eliminate the shives at the same time when the “all fibre” extraction device was used on straw batches for which the retting time was shorter, i.e., for which the pectin content was a little higher. The proportion of shives remaining trapped inside the fibrous lap was quite high then. The extra sieving step to remove shives was thus particularly required in those two cases.

Table 6. Purity of the fibrous materials in fibres during process B (i.e., for the fibre lap at the outlet of the “all fibre” extraction device, and after sieving), and number of impurities in the fibrous lap after sieving (percentage in weight).

Batch Number	After the “All Fibre” Extraction Device	After Sieving	Impurities (i.e., Shives and Dust Amount) in the Fibrous Lap after Sieving
1	50.3 ± 2.8	73.4 ± 1.5	26.6 ± 2.6
2	54.5 ±1.6	73.3 ± 1.8	26.7 ± 2.4
3	56.7 ± 2.1	76.6 ± 1.8	23.4 ± 1.9
4	63.4 ± 2.2	79.7 ± 0.9	20.3 ± 1.9

After the extra sieving step, purity values were largely increased, varying from a minimum of 73% to a maximum of 80%. The significant increase in purity as compared with that at the outlet of the “all fibre” extraction device confirmed the importance of this step for the removal of an additional fraction of shives. Both batches dew retted for 12 weeks showed only a 73% purity level, which was expected due to the low purity values observed at the Cadette machine outlet. Globally, although significantly lower than with method A with the exception of batch number 3 (Table 5), purity values of the fibrous laps after sieving should nevertheless be suitable for the further processing of the fibres into yarns and geotextiles. It is noticeable that the fibre purities in this study were much less than for those reported in a previous work from Khan et al. [16] for which the same “all fibre” extraction device was also used for extracting fibre bundles from oleaginous flax straw: 73–80% instead of 82–96%. In that study, vertical breaker rollers were implemented before the introduction of the straw to the Cadette machine. That additional operation eliminated a part of the shives even before the extraction of the fibre bundles followed by their opening.

3.5. Comparison between Methods A and B

When comparing both fibre extraction methodologies, i.e., A and B, batch number 3 (normal cut and 14 weeks of dew retting) showed a reduced purity of 73% for method A. Batches number 1 and 2 showed a minimal purity of 73% for method B, the same was also true for batch number 4 (80% purity). Batches number 1 and 2 were both retted for 12 weeks, but they corresponded to two different cutting heights, i.e., high and low, respectively. It can thus reasonably be concluded that the height of the cutting was not significant for the purity values.

In fact, fibre bundles produced using method A showed better purity as compared with method B, with the exception of batch number 3 for which the results obtained were surprisingly the opposite. This indicates the better adaptability of the breaking rollers, breaking card combination for extracting fibres bundles from oleaginous flax straws. It is thus reasonable to assume that the breaking card is less aggressive, i.e., more suitable, for these straws.

The sieving extra-step has however been of particular interest for method B as it increases the fibre purity. This step removes the smaller shives from the fibrous lap, the bigger ones, especially those entangled to the fibres, were more difficult to remove during this step. It is necessary to keep in mind that the vertical breaker rollers placed before the "all fibre" extraction device in Khan et al. [16] were not implemented in the present study; thus reducing the interest of method B with respect to fibre purity.

It is admitted that a fibre purity of more than 80% should be attained in the extracted fibre bundles to greatly facilitate the spinning step for obtaining coarse yarns. In this study, such purity level was achieved for four fibrous materials out of eight, and three of them were obtained from method A. Consequently, the latter appeared as more suitable for subsequent geotextile applications. For future work, additional improvements in fibre purity would be appreciated for other applications with higher added value (e.g., load bearing composite applications).

3.6. Fibre Bundle Lengths

The fibre bundle length of each batch was measured by randomly taking a handful of fibre bundles. Around two hundred fibre bundles were analysed per batch, and the obtained results are given in Table 7. The mean length was always more than 5 cm, meaning that all the extracted fibre bundles were suitable for the subsequent yarn production with respect to fibre bundle length.

Table 7. Length of fibre bundles (cm) inside all the produced final laps.

Batch Number	Process A	Process B
1	13.1 ± 7.1 (+75%)	7.6 ± 3.1
2	14.9 ± 7.7 (+104%)	7.3 ± 3.5
3	12.8 ± 7.0 (+68%)	7.8 ± 3.1
4	14.1 ± 7.3 (+90%)	7.5 ± 3.2

Results are presented as mean values ± standard deviations. The percentages in parentheses correspond to the relative increase in the length of fibre bundles obtained for process A in comparison with process B for the same straw batch.

In this work, the average fibre bundle lengths were largely increased in comparison with some previous results (4–5 cm) described in the literature [12]. Additionally, the length values obtained from the breaking card (method A) were improved in comparison with the results from a more recent study for which the dew retting of straw was no more than six weeks before their processing using the same equipment [16]: from 12.8 to 14.9 cm (i.e., 13.7 cm in average) instead of from 10.6 to 13.4 cm (i.e., 11.4 cm in average). It is however important to note that the values of standard deviations from the present study were increased as well, which is clearly evidencing a larger dispersion in the results around the mean value.

On the contrary, for fibre bundles extracted using the “all fibre” extraction device (method B), the lengths in this study are a little shorter in comparison with the results in Khan et al. [16]: 7.5 cm mean value instead of 8.0 cm. The most likely reason is that the moisture content inside the straw batches in the present study was 13–14% as compared with the 14–19% values at the moment of fibre extraction in Khan et al. [16]. Due to lower moisture in this study, fibres in straw batches were more rigid, and their increased brittleness resulted in more fibre breaks at the moment of their mechanical extraction in the “all fibre” device. The fibre bundle lengths for method B, i.e., from 7.3 cm to 7.8 cm, would however be high enough for further yarn processing.

In method A, fibre bundle length was much higher as it varied from 12.8 cm to 14.9 cm. As a reminder, these fibre bundles were coming from another route, i.e., horizontal breaker rollers, a thresher and then a breaking card. Such a route was thus much more favourable for linseed flax straws, constituting with no doubt a much less aggressive set-up. The breaking card was the main equipment of this route. The height and structure of its nails, their spacings and geometry were evidently more accurate for better preservation of the length of the fibre bundles once extracted. This breaking card was thus less aggressive than the “all fibre” extraction device used for the B route, hence providing a certain edge in terms of fibre bundle length. No significant difference was observed between the straw batches obtained from the same cutting height, illustrating that the dew retting duration had no real influence in this study for the mean length of the extracted fibre bundles.

As a first conclusion, an increase in the mean length of fibre bundles from 68% to 104% was found for process A as compared with process B, corresponding to an increase in length from 5.0 cm to 7.6 cm depending on the straw batch. In addition, the relative increases were more important for batches number 2 and 4 (i.e., 104% and 90%, respectively), both harvested using a low cutting height. Such a harvesting setting was thus more favourable than the dew retting duration to obtain longer bundles when using the breaking card route. The most important relative increase for fibre bundle length between the two extracting methods (i.e., 104%) was observed for batch number 2 for which the dew retting duration was only 12 weeks. The corresponding 14.9 cm mean length for the fibre bundles extracted through the breaking card route was also the most important one of the entire study, meaning that a 12-week dew retting duration was sufficient for optimizing the length of fibre bundles. As a result, extending the dew retting period by two more weeks had no positive effect on the length of the bundles obtained, and this could also be of real practical interest to the farmer who could thus free his plot more rapidly.

3.7. Elementary Fibre Tensile Properties

Figure 4 shows typical stress–strain curves, similar to the ones reported in the literature for linseed flax by Pillin et al. [17].

From these curves, the mean tensile strength and moduli as well as the failure strain are reported in Table 8. This one shows the tensile properties of the elementary fibres manually separated from the mechanically extracted bundles. Their separation from the fibre bundles was conducted with the highest care using hot water to avoid any damage. When looking at the projected diameter results, it appears that the minimum and maximum values are very different. The fibre cross-section is therefore more elliptical than cylindrical. All the batches had really different values for their minimal and maximal diameters. Indeed, the minimal diameter ranged from 10.8 to 12.3 µm, whereas the maximal one ranged from 20.9 to 24.7 µm. Values for the cross-sectional areas were between 181 and 242 µm^2, and this would correspond to diameters ranging from 15.2 to 17.6 µm if considering the cross-section of all fibres as an ideal cylinder. The diameter values obtained for these elementary fibres were in the same order of magnitude as those obtained by Ouagne et al. [12] and Khan et al. [16]. So, it is believed that the manually extracted fibres were indeed individual ones.

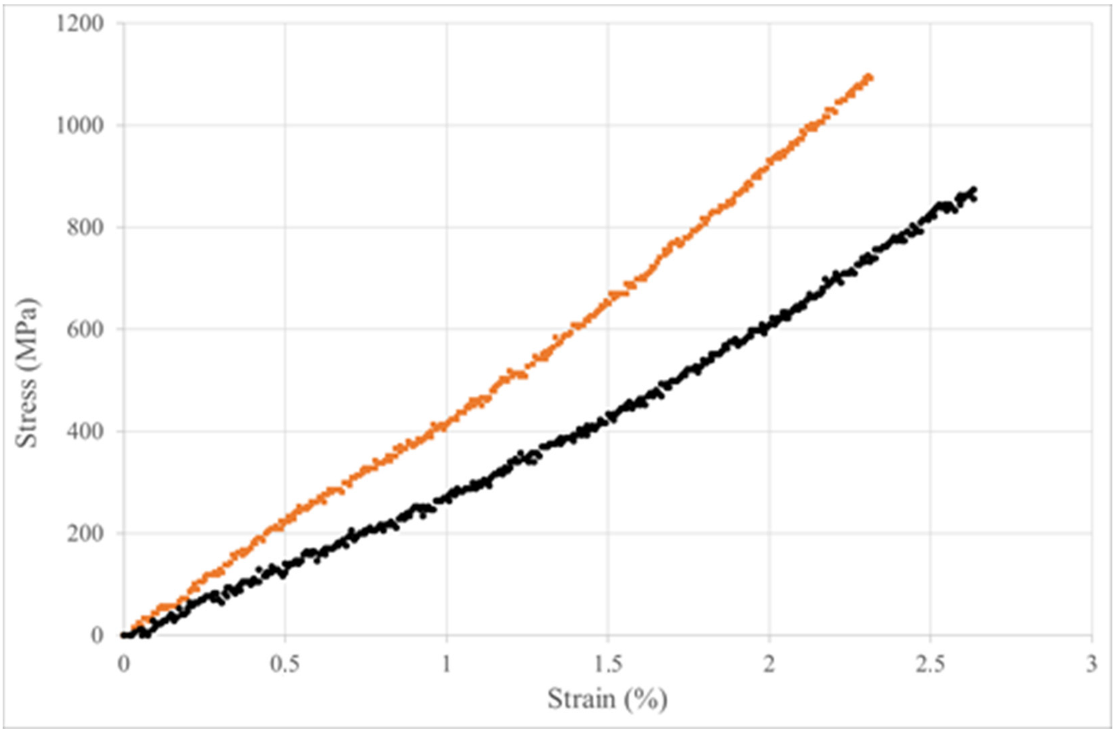

Figure 4. Examples of typical linseed flax stress–strain curves used to determine tensile properties of single elementary fibres.

Table 8. Tensile properties, minimal diameter, maximal diameter and cross-section area of individual fibres coming from the four studied batches and tested after extraction, using both methods A and B.

Characteristic	**1A**	**1B**	**2A**	**2B**	**3A**	**3B**	**4A**	**4B**
Tensile strength (MPa)	922 ± 364	737 ± 319	781 ± 326	759 ± 309	855 ± 348	788 ± 450	820 ± 302	807 ± 314
Young’s modulus (GPa)	56.5 ± 19.6	53.4 ± 14.8	53.3 ± 18.2	53.4 ± 17.2	52.3 ± 17.2	48.6 ± 16.5	52.9 ± 12.0	51.2 ± 14.3
Strain (%)	1.7 ± 0.6	1.6 ± 0.5	1.6 ± 0.5	1.6 ± 0.6	1.6 ± 0.5	1.5 ± 0.5	1.7 ± 0.6	1.6 ± 0.5
Minimal diameter (µm)	11.6 ± 2.0	10.9 ± 2.9	12.2 ± 2.9	12.3 ± 3.6	10.8 ± 2.5	11.8 ± 2.8	11.9 ± 3.0	12.2 ± 3.3
Maximal diameter (µm)	22.0 ± 5.0	22.5 ± 6.1	23.0 ± 4.8	23.7 ± 6.1	22.1 ± 4.0	24.7 ± 7.4	20.9 ± 4.2	23.4 ± 5.2
Cross section area (µm^2)	181 ± 76	204 ± 94	230 ± 92	242 ± 124	195 ± 78	241 ± 128	201 ± 82	238 ± 114

The tensile strength values varied from 737 to 922 MPa. The minimal one (i.e., 737 MPa) was obtained for batch number 1B, which corresponded to the fibre bundles extracted from the straw batch number 1 using the B methodology. For its part, the maximal value (i.e., 922 MPa) was obtained from the same straw batch but with fibre bundles extracted using the A methodology (i.e., batch number 1A). As these two opposite tensile strength values came from the same straw batch (high cut and 12 weeks of dew retting), these results clearly highlight the interest of method A. Indeed, in addition to better preserving the length of the extracted fibre bundles (Table 7), the breaking card route also led to more mechanically resistant elementary fibres (+25% for tensile strength in the case of fibres extracted from straw batch number 1). In parallel, the Young’s modulus was also 6% higher for the 1A individual fibres, i.e., 57 GPa instead of 53 GPa for the 1B ones. Evidently, the breaking roller and breaking card combination was less aggressive at the moment of the fibre extraction. Additionally, when comparing all the results each other independently of both the method of obtaining the straw batch and the fibre extraction method, Young’s modulus values varied from 49 GPa (batch number 3B) for the minimal value to 57 GPa (batch number 1A) for the maximal one. Thus, the elementary fibres with the higher tensile strength value (i.e., the 1A ones) were also the stiffer ones. When looking at the average values for tensile strength and Young's modulus for methods A and B, they were 845 MPa and 773 MPa, respectively, for tensile strength, and 54 GPa and 52 GPa, respectively, for Young's modulus. So, method A was more suitable as compared with method B with regard to the elementary fibre's tensile properties, surely making the breaking roller and breaking card a less severe (i.e., less aggressive) extraction device in comparison with the "all fibre" machine.

When considering the two different dew retting durations, the average values for tensile strength were 800 MPa and 818 MPa, respectively, for 12 weeks of dew retting and 14 weeks of dew retting. Similarly, Young's modulus values were 54 GPa and 51 GPa, respectively. Although the 12-week dew retted batches showed slightly lower average values for tensile strength, the difference was particularly limited. In parallel, elementary fibres corresponding to this shorter retting time were a little more rigid. This could be the consequence of a sufficient and optimal dew retting duration at 12 weeks. In conclusion, two additional weeks of dew retting had no real interest for the tensile property.

For yarn production, the choice has thus been made to use the 1A fibre bundles, i.e., bundles extracted from straw batch number 1 (12 weeks of dew retting) using the breaking roller and breaking card route, as the fibres show longer mean length (i.e., 13.1 cm) and the highest tensile properties in the entire study.

3.8. Yarn's Tensile Strength

Yarns were successfully produced from the 1A fibre bundles using a gill-flyer spinning machine, and the characteristics of the optimal 100% linseed flax twisted yarn obtained are presented in Table 9. In particular, the tensile strength value was 49.4 MPa. This optimal yarn was manufactured by optimising the machine parameters, e.g., the speed of the feed rollers, the speed of the delivery rollers, etc. The spindle speed was important to maintain a good twist level for yarn manufacturing. It is also important to note that by increasing the twist level, the linear mass values of yarns were increased. Increasing the twist level brings the fibres in close contact together and helps to increase its specific weight (i.e., its weight per unit of length). The optimal yarn, whose characteristics are presented in Table 9, corresponded to 100 twists per meter, resulting in a linear mass value of 4060 tex.

Table 9. Characteristics of the optimal 100% linseed flax yarn, i.e., corresponding to 100 twists per meter.

Characteristic	**Diameter (mm)**	**Linear Mass Tex**	**Breaking Force (N)**	**Tensile Strength (MPa)**
Value	2.5 ± 0.3	4060 ± 132	242.5 ± 49.1	49.4 ± 10.0

Results are presented as mean values ± standard deviations.

As compared with previous results in the literature, the tensile strength of the optimal yarn showed a real interest of the linseed flax fibres for yarn manufacturing for geotextile applications. In Ouagne et al. [21], hemp and coir yarns used for geotextile applications had a tensile strength of 45 MPa and 14 MPa, respectively, for linear mass values of 4312 tex and 6172 tex, respectively. The results of the optimal linseed flax yarn are much higher than those of the coir yarn (+250% for tensile strength), which is already used in geotextile applications. The optimal linseed flax yarn in this study was also 0.66 times finer than the coir one. So, fewer fibres enabled better strength results when using the linseed flax fibres to produce yarn for geotextile applications. The linseed flax yarn's tensile strength and linear mass value were in the same order of magnitude as those of the hemp yarn in Ouagne et al. [21]. The yarn in this study will thus undoubtedly be usable for subsequent geotextile applications. It was however less resistant (−31% for tensile strength) than the textile flax yarn having an equivalent linear mass value and also presented in the study mentioned above (72 MPa tensile strength and 4187 tex value), illustrating the reduced mechanical resistance of the oleaginous flax fibres in comparison with that of the textile flax ones.

It is also important to note that a significant decrease in the standard deviation of the yarn's tensile strength was observed as compared to the results obtained for the elementary fibres. Indeed, for the 1A elementary fibres, the standard deviation for tensile strength represented 39% of the associated mean value (Table 8), and it was only 20% in the case of the optimal linseed flax yarn (Table 9). This decrease in the standard deviation may be due to the assembly of the same yarn of different fibres with dispersed mechanical properties which come together to form a more homogeneous and mechanically uniform yarn thanks to the twist, resulting in a reduction in the tensile strength's standard deviation.

3.9. Yarn's Observation with a Microscope during the Durability Tests

Before testing the yarns in real soil conditions, the latter were coated with chitosan or linseed oil. Images of the three different yarns (i.e., non-coated yarn and yarns coated with chitosan or linseed oil) were taken with a microscope, and they are presented in Figure 5. The coating on the yarns can be clearly evidenced when applied. Page et al. [22] showed that linseed oil coating on textile flax fibres gave a glossy appearance to them when observed with a microscope, and the same was true in the present study for linseed flax fibres.

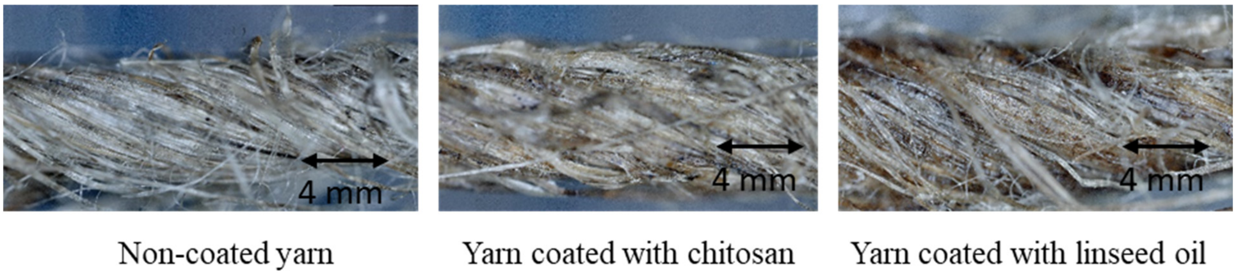

Figure 5. Microscopic representation of a non-coated yarn and yarns coated with chitosan or linseed oil.

In the present study, it was visually observed that after chitosan coating and water evaporation in the ventilated oven, the yarns became more compact. Some form of stiffness was also observed in those yarns, with the evaporation of water used to dissolve the chitosan prior to its application, leading to the formation of a crust on the yarn surface. Linseed oil coated yarns also became more compact. However, no increase in the stiffness of these yarns was observed, which will be an advantage for handling and laying the geotextiles. Overall, the weight of both types of coated yarns was found to be increased after coating. This confirmed that there was indeed some coating on the yarns. The moduli of the different yarns, before any kind of ageing, increases from 711 ± 92 MPa for non-coated to 917 ± 115 MPa for linseed-oil-coated and 954 ± 136 MPa for chitosan-coated. The moduli values of yarns significantly rise between the non-coated and coated ones (either by chitosan or linseed oil). This shows that both coatings increase the stiffness of the yarn by creating a composite-like shell at the periphery of the yarn.

Microscopic images of the yarns were also taken during soil durability tests after two and four months (Figure 6). These images showed that there was some kind of biodegradation over time, and the latter was perfectly visible. The shape of the yarns was clearly affected due to this biodegradation. For the non-coated yarn, the microbial attack occurred quite rapidly, and the microorganisms in the soil "ate" a large portion of the yarn; the latter was broken into small pieces after only two months. On the other hand, yarns coated with chitosan and especially with linseed oil were able to maintain their initial shape longer, although their strength was found to be decreased due to biodegradation. Yarn coated with chitosan was also broken into many small pieces but only after 2.5 months.

For its part, the yarn coated with linseed oil kept both its shape and its integrity for a much longer period, i.e., until month 4, but its integrity finally disappeared at the end of month 5. Indeed, at that moment, only two yarns coated with linseed oil out of four were found in the same shape, and the two others were broken into two pieces. Conversely, non-coated and chitosan-coated yarns were found in several pieces from month 2 and month 2.5, respectively, and measuring their tensile strengths was completely impossible after month 4.

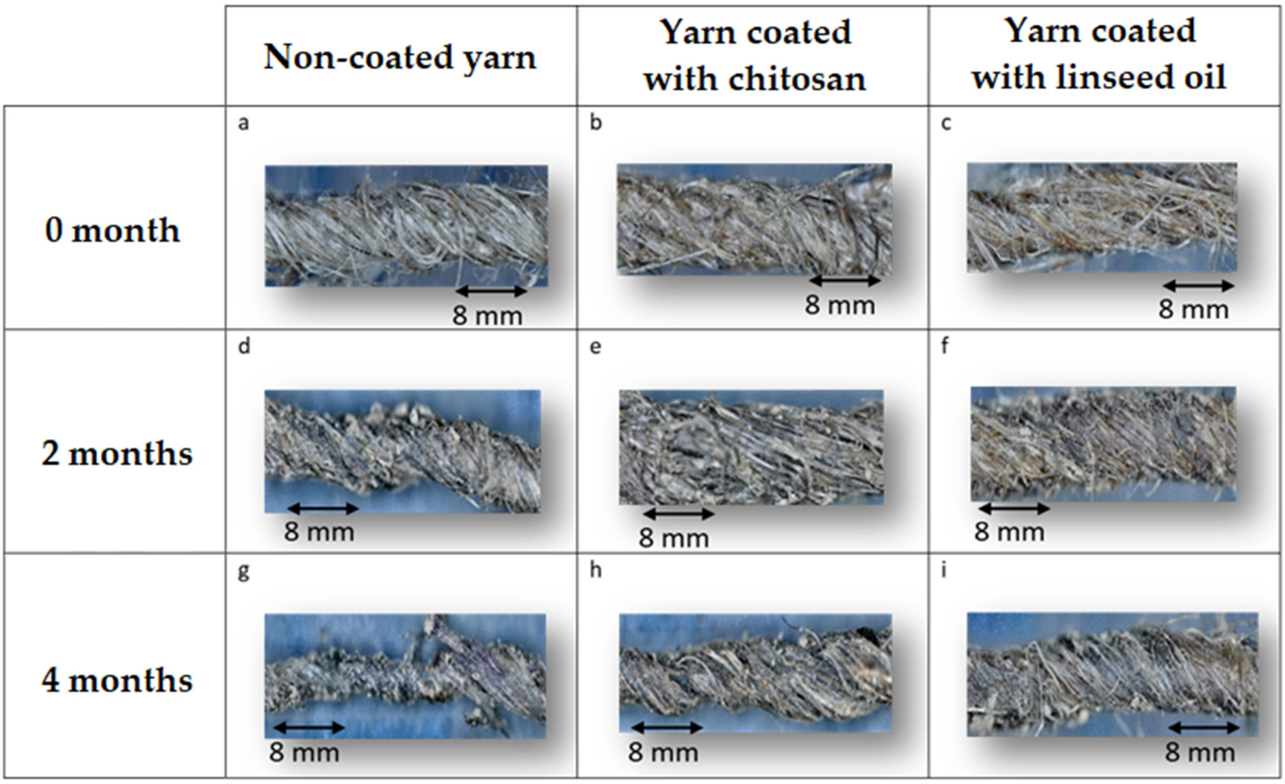

Figure 6. Microscopic representation of non-coated yarns after 0 (**a**), 2 (**d**) and 4 (**g**) months in the soil, yarns coated with chitosan after 0 (**b**), 2 (**e**) and 4 (**h**) months in the soil, and yarns coated with linseed oil after 0 (**c**), 2 (**f**) and 4 (**i**) months in the soil.

The yarns coated with linseed oil were the only ones for which the shape was more or less maintained until the end of the durability tests, although it became more and more fragile as the microorganisms in the soil progressively degraded it over time. It is however evident that their activity was largely slowed in comparison with the untreated yarn and the one treated with chitosan. The "hydrophobation" of the surface of the yarn coated with linseed oil is a priori the main reason for that.

When looking at the microscopic images of the yarns in Figure 6 at different times during the soil durability tests, it is possible to conclude that the biodegradation of fibres occurred for the three tested yarns, leading to its breaking. Soil particles were also visible in between the fibre bundles after a while, which will certainly increase the kinetics of biodegradation. However, it is obvious that the linseed oil coating treatment applied on the 100% linseed flax yarn slows down this kinetic.

3.10. Yarn's Tensile Strength Evolution during the Durability Tests

The tensile strength of the non-coated yarn and yarns coated with chitosan and linseed oil was measured during the durability tests. The results obtained are presented in Figure 7. Each yarn was also tested for tensile strength before it was buried in the soil. This initial tensile strength value was the reference value, and the samples were tested again after specific durations. Before testing the samples, they were taken out of the soil and placed on a grid at atmospheric conditions for 24 h so that they could dry. The extra-fine soil particles were then removed with care before mechanical testing. Here, it has to be mentioned that the slight reduction in the tensile strength observed before the beginning of the durability tests for both coated yarns (i.e., 45.6 MPa and 41.0 MPa, respectively, for chitosan and linseed oil) is mainly the consequence of the increase of their diameters, due to the application of this coating treatment: 2.93 mm and 2.70 mm for the yarns coated with chitosan and linseed oil, respectively, instead of 2.5 mm for the non-coated yarn (Table 9).

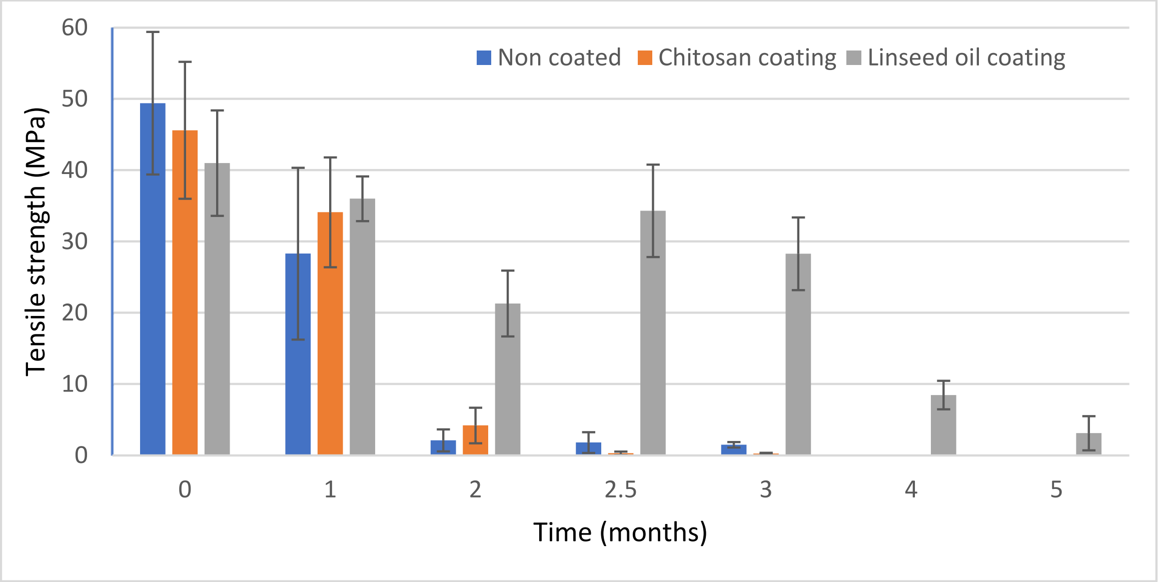

Figure 7. Tensile strength of non-coated and coated yarns as a function of the duration of time the yarns have been in the ground.

After month 1, the tensile strength of the non-coated yarn was decreased by 43%, which was the worst result at this time. For chitosan-coated yarn, the decrease in tensile strength was 25%, and it was only 12% for the yarn coated with linseed oil. So, it was evidenced that the non-coated yarn showed more loss in tensile strength as compared with the other two yarns. Lignin content and the crystallinity rate of cellulose are known for their benefits in limiting the kinetics of biodegradation in soils [28]. Lignin content for coir fibre is around 35%, which is very high and makes it quite resistant to microbial attack. Jute has around 12% lignin content, whereas other bast fibres (e.g., hemp or flax) have much lower lignin content (less than 5%). In our case, lignin content was 3.6% (Table 4), which was very limited as compared with jute and coir fibres. So, due to low lignin content, non-coated yarns were rapidly subjected to microbial attack and lost their strength very quickly. Conversely, chitosan is known for its antimicrobial properties [29,30], thus explaining why the loss in tensile strength after month 1 was less significant (25% instead of 43%) for the yarn coated with chitosan. The yarn coated with linseed oil was, however, the most promising one, probably due to the hydrophobic behaviour of linseed oil [31]. So, if the water in soil is repelled during the durability tests, there is then definitely less risk of microbial attack of the fibrous yarn.

At month 2, the tensile strength values of the yarn still continued to decrease, and this was also clearly evidenced by visual observation (Figure 6). In particular, the non-coated yarn showed a 96% loss in its tensile strength, thus confirming the observations already made after month 1. The same trend was also observed for the chitosan-coated yarn for which the tensile strength was 91% less than the initial value. Although chitosan has known antimicrobial properties, it is soluble in acidic conditions. The pH of the soil for the durability tests in this study was estimated to be around five, meaning that it was slightly acid. Thus, it is reasonable to assume that coated chitosan was progressively dissolved during the test, probably being removed in totality from the yarn's surface after two months, explaining the tensile results equivalent to the ones of the untreated yarn at this time. Meanwhile, the yarn coated with linseed oil was still more resistant, i.e., less than 50% reduction in its tensile strength even though the mechanical strength value at this time was surprisingly rather low compared with the other values measured during the durability test (Figure 7). Linseed oil firstly reduced water diffusion inside the coated yarn and, in a second phase, this prevented the attack from microorganisms.

After month 3, the non-coated and chitosan-coated yarns lost almost 97–99% of their initial tensile strengths as shown in Figure 7. It was no longer possible to assess their tensile strengths at month 4 and the following times. On the contrary, the yarns coated with linseed oil were able to maintain their shape for a much longer time and some strength until month 5. The reduction in the initial tensile strength for that yarn was 16%, 31%, 79% and 92% after months 2.5, 3, 4 and 5, respectively. Finally, tensile strength values were not measured after month 6 and month 8 as the yarns were all degraded, including the one coated with linseed oil.

In conclusion, non-coated yarns are almost completely degraded in soil after only two months. In parallel, it is difficult to conclude here on the interest of coating the 100% linseed flax yarn with chitosan. Certainly, such a coated yarn should not be used in acidic soils. It should therefore be reserved for neutral soils or especially basic ones (i.e., calcareous soils), even if durability measurements are conducted for future work in such pH conditions. Conversely, yarns coated with linseed oil showed a significant resistance against biodegradation over time, even after five months. It can thus be concluded that yarns coated with linseed oil are good candidates to resist to the microorganisms in soils, and this would increase the service lives (up to about five months) for geotextiles made from these treated yarns.

4. Conclusions

Linseed flax is an important crop due to interest in its seeds. This study evidenced that the straw could be valorised too if no shortener is used. The presence of bast fibres in straw is in the range from 29–34%, making linseed flax an interesting complementary crop for providing natural fibres (up to 630 kg/ha in this study). Before the fibre extraction step, dew retting is an important process as it helps to better preserve fibre bundle length while favouring their dry mechanical extraction. It was witnessed in the study that with increased dew retting durations, cellulose content inside fibres simultaneously increased with a decrease in the pectin content, which is an advantage for the subsequent mechanical extraction of fibres.

Fibre purity is mainly dependent on the extraction method, and the best purity in this study (86%) was obtained with horizontal breaker rollers followed by a thresher and then a breaking card from a batch dew retted for 14 weeks in the southwest of France, which has dry summers in comparison with Normandy, for example, which is a more favourable territory. Fibre bundle length was then in the range from 13–14 cm, which is interesting for further spinning stages. The mechanical properties of elementary fibres manually extracted from these bundles were also promising with tensile strength and Young's modulus of 922 MPa and 56 GPa, respectively.

One hundred percent linseed flax yarns were manufactured from the optimal bundles using a gill-flyer spinning machine. With a tensile strength and linear mass value of 49 MPa and 4060 tex, respectively, they were then assessed for durability tests in the soil. Non-coated yarns and those coated with chitosan lost their tensile strength up to 96% and 91%, respectively, after only two months, whereas yarns coated with linseed oil lost only 31% tensile strength after three months. Due to the hydrophobic behaviour of linseed oil, using it for coating significantly increased the service life of linseed flax yarns. Such post-treatment should be preferred to increase the geotextile service life.

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