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Related with the research line

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General Introduction

Textiles have been an integral part of human daily life for thousands of years and has evolved with human history. Climate change is one of the major challenges facing humanity today. European consumption of textiles has, on average, the fourth-highest impact on the environment and climate change. For several years, the textile industry has worked on reducing its dependence on oil production and its impact on the environment, in particular the water pollution and greenhouse gas emissions. Several initiatives are emerging at the European level to achieve sustainable and circular textile management and launch strategies to change textile production and consumption. A large part of the environmental impact comes from material resources. The two main sources of textiles in the world are synthetic fibres and cotton. Synthetic fibres, such as polyester, are derived from petroleum and disperse microplastics in the environment while cotton fibres require large amounts of water, pesticides and fertilisers. Another solution for accessing to more responsible materials is the use of textile flax. However dedicated spring textile flax cultivation area has reached its maximum potential in France and this production is no longer sufficient to meet the always increasing market demand. The price of textile flax fibres has increased threefold in three years because of this very high demand and the difficulties to grow textile spring flax due to climatic changes. It is in this context that hemp is positioned to supply the European textile market in ecological, durable and local fibres as a complementary resource to textile flax. Over the past few years, an increasing number of research articles and reviews focused on reporting on its ecological virtues and the ability to produce high quality textile fibres from the hemp plant. A high potential for reliable local textile hemp fibre production is suggested in Europe. Actually, hemp was a major crop in the past, but the know-how has been in a great extent lost, and the techniques and mechanisation of olden times are no longer adapted to today's lifestyle.

The development of complete textile hemp value chain starts by the comprehension of the needs and the issues in terms of mechanisation and understanding the hemp responses to its environment. The area of Normandy, where a large part of textile flax is cultivated, is considered highly suitable for textile hemp production. On the one hand the climatic conditions are favourable for the cultivation and, on the other hand, the flax-processing equipment is already present in this area. Due to the similarity between the two plants, hemp could be processed with the same equipment. Investigations for suitable parameters as well as the development of new equipment are necessary to complete a new value chain. Another obstacle is the lack of available varieties dedicated to the territory and the textile application. For the moment most varieties on European seed catalogue are suitable for harvesting seeds and biomass for other applications. Genetic research is an essential element in the development of an industrial textile hemp crop, as it has been the case for the improvements of other crops. The enhancement of specific varieties with high fibres content and textile grade quality would support the development of the textile hemp chain value. In this context, the water agency “l’Agence de l’Eau Seine-Normandie” supports the launch of the hemp textile industry in Normandy and the research work of this thesis.

This manuscript is structured in four parts in a way to provide elements of knowledge on the properties of hemp fibres and their behaviour during their transformation process.

The first part provides a state of the art on the hemp plant and the relevant background for the work exposed in this thesis. A general description of the hemp plant and its history explains the evolution of hemp cultivation and its renewed interest. Key findings regarding the properties of hemp fibres and the relationship with the agronomic practices are reviewed. A presentation of the elements of hemp processing for textile application is exposed.

The second chapter presents a study on the varietal selection of hemp to obtain textile quality fibres. Several varieties provided by seed breeders are analysed and experimental work over three years was developed to understand the variation of properties between the varieties.

This work provides indications to the seed breeders for selection optimisation and the development of varieties adapted to the terroir and the textile application.

The third chapter is devoted to the industrial development of hemp fibre production. The evolution of the mechanical harvesting of hemp, which currently requires a specific mower, was followed, and the study of scutching parameters to enhance the fibres extraction from the plant was conducted, as well as an economic approach.

The last chapter addresses the spinning processes. The textile long fibres are firstly studied regarding their wet spinning potential. Another solution to spin hemp yarns from by-products was investigated: non-aligned hemp and linseed flax are explored.

The manuscript ends with a general conclusion and perspectives paving the way to new areas of research and understanding.

Chapter 1.
The State of the Art

1.1. Description and Uses of Hemp

1.1.1. The Cultivation of Hemp

1.1.1.1. Historic Uses of Hemp Fibres

Hemp is a multipurpose crop that has been grown for centuries. It was mainly used for its protein-rich seeds, and its fibres, in particular for the textile industry, for sailing equipment or for papermaking *(Bouloc, 2006)*. It is one of the oldest plants domesticated by humans. Central Asia is believed to be the cradle of domesticated hemp with the evidence of clothing woven from hemp dating back to 6,200 BC as well as ropes or samples of paper dating back to 3,000 BC found in China *(Hillig, 2005)*. In Japan, hemp was used to create the most precious clothing, from summer kimonos to samurai attire and the garments of a Shinto priest. Every aspect of work involving hemp, from planting to weaving was women's labour *(Hash Marihuana & Hemp Museum, Barcelona)*.

Hemp cultivation expanded to Western Asia, Egypt, Russia and East Europe and became widespread in Europe from 500 BC *(Chevalier, 1944)*. The methods employed to achieve finished hemp clothes in Asia and Europe diverge significantly, resulting in two approaches for the extraction of bast fibres and for the weaving *(Clarke, 2010)*. In 1456, Gutenberg published the first printed Bible on hemp paper. Hemp played an important role during the discovery of America by Christopher Columbus: the sails and ropes of his ships were made from hemp; the gaps between the planks were caulked with hemp to waterproof the ships; hemp seeds were used as protein-rich food for the crew and also meant to be planted in the newly discovered lands; hemp oil was also used in oil lamps on ships. Hemp cultivation reached its maximum expansion between the 16th and 18th centuries with the strong demand for maritime countries for the ropes necessary for warships *(Bouloc, 2006)*.

Hemp cultivation experienced a first decline at the beginning of the Industrial Revolution with the arrival of the steam engine and the development of the wood-based papermaking process *(Brouard, 2010)*. The distribution of the hemp cultivated area in the 19th century is illustrated in Figure 1.1.

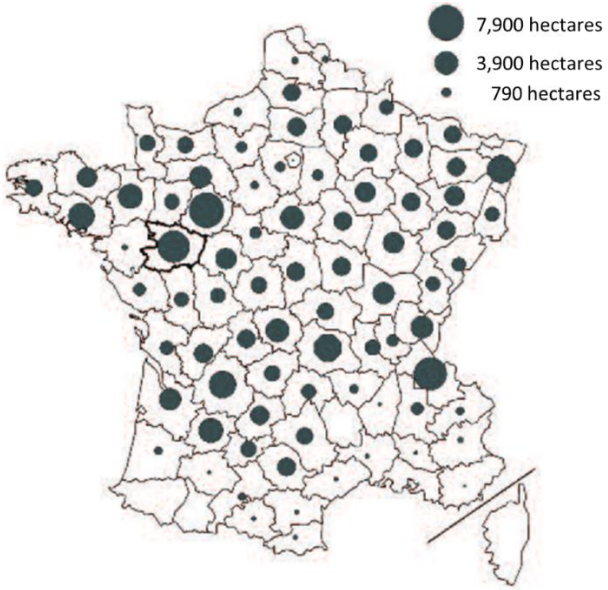


Figure 1.1: Hemp cultivated areas in France in 1838 (Brouard, 2010).

Nevertheless, the high declined of hemp production happened in the middle of the 20th century. It suffered from the development of synthetic fibres, such as nylon, the competition with cotton, the reduction in sailing boats, the labour crisis and anti-drug policies due to its connotation with narcotics *(Lévêque, 1925)*. In 1937, The « Marijuana Tax Act" banned the hemp cultivation in the USA *(Fike, 2016)*.

As a result, most of the genetic diversity of European hemp was lost and breeding programmes, as well as mechanisation, were drastically reduced *(Salentijn et al., 2015)*. In the midst of the Cold War, Soviet bloc countries such as Poland, Romania, Hungary retained their expertise in the production of textile hemp. The cultivation of hemp for long line technical fibres was still carried out until recently in the countries of Eastern Europe due to the lower labour cost *(Sponner et al., 2005).*

Long-time forgotten and associated with the use of cannabis, hemp experienced a revival in the 20th century, greatly due to its environmental performances. From oil to fibre, the plant is used in all its forms in textiles, food, construction, cosmetics and health. Although the demand for textile hemp is increasing, no large-scale production of high-quality hemp yarns and fabrics are settled in Europe. The whole process chain is not yet optimised, and the producers cannot yet supply homogenous, high-quality hemp fibres that the spinning companies require. The most promising way to produce high-quality hemp yarns involves long hemp fibres. The chain of processes to obtain those valuable fibres consists in growing with adapted agronomic choices the hemp, cutting parallelise and align stalks on the ground for dew retting. The parallelised stems after retting are baled dry and brought for fibre extraction using scutching and hackling before wet-spinning.

1.1.1.2. Methods of Harvesting and Processing

A challenge faced at industry level until recently is the lack of availability of machinery for the harvesting and processing of hemp at each level. As recent hemp market was mainly focused on the extraction of medicinal components and short unaligned fibres, the hemp crop harvesting and processing machinery mainly aims to harvest and process crushed stalks, seeds and untangled fibres. Different types of machinery has been developed such as hemp piecewise harvester of stalks, double-cutting harvesters of hemp, and drum-type hemp cutters *(Pari et al., 2015).* However, they have disadvantages of high waste rate of fibre and high breakage rate and they cannot suit for harvesting long fibres as required by the textile industry *(Müssig, 2010)*.

The key points to be developed for harvesting hemp long fibres for the use of flax scutching lines are: (1) an efficient cutting element with an adjustable head height design in accordance with the characteristics of the hemp stems in order to harvest the maximum amount of valuable fibre, (2) a conveyor mechanism that reliably sends the hemp stems out in time according to the feed quantity of cut hemp stems, and (3) lay down the stems aligned and parallel without damaging them *(Pari et al., 2015)*. The case of lodging is rare in hemp, so it should not hamper much harvesting. As soon as the hemp stems are harvested aligned in 1 metre lengths and arranged in a parallel way, it would be possible to use the flax dedicated machine for further processing such as returning the stems while retting and collecting them by balers forming cylindrical bales commonly used in the flax value chain.

In the late 2010s, new harvesting machines developed in China or Italy (BCS Group, Abbiategrasso, Italy), appeared on the European market. They cut the hemp stems at desired height and drop off the stems in parallel and aligned manners. This type of machine holds a single sickle bar, then the entire length of the stems is laid on the field. To separate the stems

into two portions of one metre, a cutting disc machine is used in succession. Machines are presented in Figure 1.2.



Figure 1.2: Single sickle bar mower machine (A-B) and a cutting disc element (C).

This type of machine is, however, not fully adapted for large-scale, efficient harvesting but they are more designed for small-scale production. The course of mechanisation for large-scale production is progressing in the recent years and large improvements on mechanisation have been made. New machines, more efficient and adapted to large-scale harvesting, are being developed, for example by Hyler company or Cretes Valtech Group, both from Belgium.

1.1.1.3. Retting

The first step after harvesting to produce long hemp fibres is retting. Retting consists in removing the pectic substances located in the middle lamella that glue the fibres together and to the woody core of the hemp stem. This step is important and essential to obtain fibres bundles with great potential of divisibility necessary for the spinning step *(Kozłowski et al., 2020).* After retting, the stems must dry to a suitable moisture content to stop the retting process before storing without risk of deterioration. This drying process involves internal stresses that helps the fibre bundles in the stem to contract and partially detach from the woody core *(Horne, 2020).*

Water-Retting

In the past, hemp was water-retted, meaning that plants were submerged in water for a period of time (Figure 1.3). It generally remained one or two weeks in the water *(Bonnétat, 1919)*. This resulted in great uniformity and quality long fibres. It was retted in a watercourse, or in stagnant water. Retting with stagnant water led to faster results but poorer quality of fibres compared to running water.

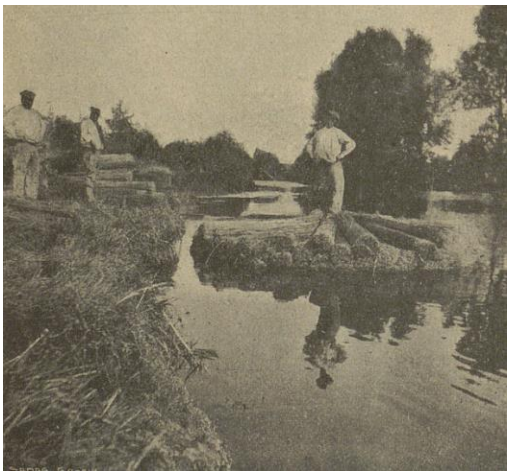


Figure 1.3: Water retting in Indre, France (Lévêque, 1925).

This process was prohibited in most countries of Europe from the 18th century as it causes water pollution releasing organic acids, other fermentation products and nauseating odours *(Brouard, 2010; Jarrige, 2013).*

Dew-Retting

Even though, East-European countries were still using water-retting until recently, dew-retting is now preferred in Europe. Stems are left spread out in the field after harvesting and biodegradation of pectin substances is achieved by micro-organisms from soil that favours the separation of the bast fibres. This approach has low environmental and economic impact but is highly dependent on climatic conditions *(Turunen, 2006)*. The control of this process is therefore difficult and it has a high influence on fibre quality *(Bleuze et al., 2018; Mazian et al., 2019; Réquilé et al., 2021).*

Other Retting Processes

Another process is stand-retting. In this case, the plant is left standing in the field so that they grow until the senescence *(Assirelli et al., 2020).* Some alternatives to the previously described field retting processes have been attempted such as enzymatic treatment, chemical separation, steam explosion or other physical process *(Manian et al., 2021; Grégoire et al., 2019; Sauvageon, 2019; Riddlestone et al., 2006; Thomsen et al., 2006; Garcia- et al., 1998).* However, these techniques are either not sustainable or expensive and difficult to industrialise.

1.1.1.4. Methods of Extraction

The purpose of the extraction stage is to separate the woody core of the hemp stem from the fibres and completely remove the woody core to obtain the fibres.

Manual Extraction

The extraction of fibres is commonly achieved by mechanical processing although historically the extraction was performed with manual machines *(Horne, 2020)* as presented in Figure 1.4. The fibres were detached from the hemp shives by crushing and hand-scutching. Crushing was done using an articulated easel and scutching was done by hand and used for the strongest stems. The hemp was then combed, with a large iron comb, and presented in the form of a handful of a dozen pounds *(Brouard, 2010).*

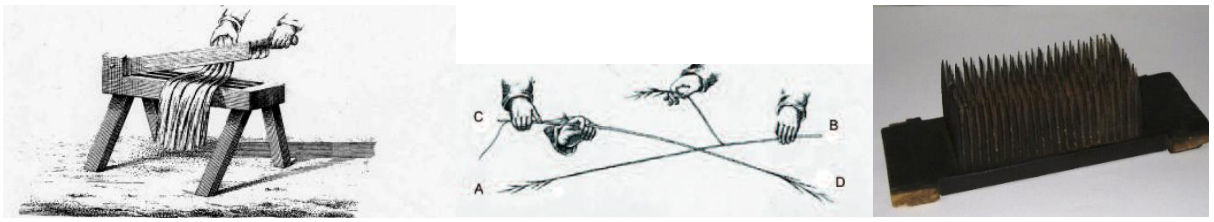


Figure 1.4: Crushing system, hand scutching and an iron comb (Brouard, 2010).

Mechanisation

The development of a mechanical system of extraction began in the 18th centuries when arose sanitary problems with retting. A system, called machine of Christian was designed to crush the stems and remove the hemp woody core *(Jarrige, 2007)*. Other systems were then developed, as shown in Figure 1.5. The stems are crushed to clean the fibres and remove the remaining shives.

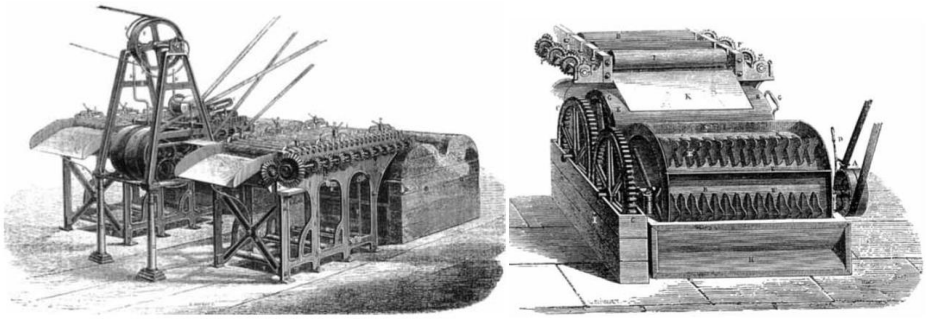


Figure 1.5: Crushing and scutching machines in Vangelieu (Oise), France (Barral, 1864).

Before the extraction the roots were removed with a root-cutter machine (Figure 1.6) so that the stems were deprived of this ligneous part which does not present valuable fibres *(Barral, 1864).*

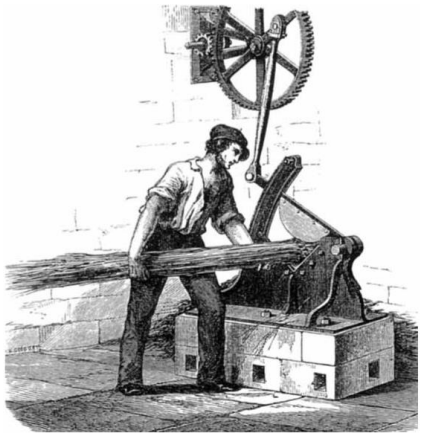


Figure 1.6: Roots cutting system in Vangelieu (Oise), France, Août 1863 (Barral, 1864).

More recently and until now, the fibres are extracted using mechanical approaches such as scutching or decortication. The quality of the fibre required by the end market and application determines the choice of the extraction method.

Scutching Machines

The scutching (by reduction the combination of crushing and beating) process is generally used when high-quality fibres are targeted. The method is similar to the one used for the production of long flax fibres and very similar to the mechanism presented above. With this method, the harvested stems have to be kept aligned and parallel. First is the breaking stage: the stems are mechanically crushed and broken along their length by a series of fluted rollers, either horizontally or vertically arranged. During this breaking/crushing process, the woody core is detached from the fibres. The efficiency of this stage is highly dependent on the degree of retting of the hemp plant. Secondly is the scutching stage: all the remaining woody core pieces adhering to the plant fibres are removed by a system of turbine blades. The fibres pass through a beating system that operates first on one side of the fibres and then on the second side. The main output of these processes is the long-aligned fibres. However, some fibres generally break and fall into the waste. Those fibres are separated from the woody core and other impurities by passing through some cleaning units. They are called tow fibres. The greater the degree of retting, the easier the extraction and the higher the yield of long line fibre and the lower the yield of tow fibre of total processed fibre *(Horne, 2020).*

Other Decortication Systems

Another extraction method is decortication. The principal method used to conduct a decortication operation is hammer mills. It consists in cutting the hemp stems with a guillotine or bale cutter to decrease the length of the stems to ensure efficient feeding. Hammer mills itself consists of a cylinder equipped with beaters that rotate at high speeds and decorticate the hemp stems. It is fed vertically into the top of the cylinder.

The use of breaker rollers is another method to conduct decortication. This system allows the rollers crush the hemp stalks via circular motion. For example, in Wang et al (2018), the device is arranged in two opposite orientated rows of 4 rollers, with counter rotating, helical geared and involute profiles (S. Wang et al., 2018).

The decorticated fibres are much less suited for the manufacture of textiles than scutched fibres but are well suited for the production of technical fibres. The decortication generally causes mechanical damages to the fibres limiting their mechanical potential and therefore their application spectrum *(Horne, 2020; S. Wang et al., 2018).* Decortication is commonly used for the extraction of fibres from plants stems without retting process (Kozlowski et al., 2004).

1.1.2. Hemp Production

1.1.2.1. The Attractive Aspect

The Advantages of Hemp Plants

Since the beginning of the 21st century, hemp cultivation knows a resurgence of interest. Its ecological and versatile aspect makes it particularly attractive and an interesting alternative to the two main current sources of textile fibres: cotton and synthetic fibres which are associated with major environmental problems *(Ellen MacArthur Foundation, 2017).* Synthetic fibres deplete fossil energy and growing cotton requires a lot of water, fertilisers and pesticides, unlike hemp *(van der Werf and Turunen, 2008)*. Indeed, because of its qualities of resistance and rapid growth, hemp does not currently require any (or low) pesticides or insecticides. It can cover the field surface and hamper the development of undesirable weed.

It needs less water than common rotational crops such as wheat, sugar beets or corn *(Dewey, 1913)*. Cotton requires 2.5 times more water than hemp per hectare of cultivated land *(Duque Schumacher et al., 2020).* In addition, hemp fits easily into the crop rotation cycle, it is a good head of rotation in organic farming, helping to increase soil fertility and soil structure and could also be used in phyto attenuation strategy to value contaminated land (De Vos et al., 2022; Linger, 2002; Mańkowski et al., 2020; Pudełko et al., 2021, 2021; Turunen, 2006; Zimniewska and Romanowska, 2022).

The Advantages of Hemp Fibres for Garment Textiles

The hemp fibres have numerous advantages for textile applications (Kozlowski and Muzyczek, 2023; Zimniewska and Romanowska, 2022)*:*

- It has a high hygroscopicity and fast moisture transport. Hemp is superior to cotton regarding moisture absorption and dissipation. The values for hemp are almost twice as high in comparison with cotton. This absorbency rate is also beneficial for dying processes, where hemp fabrics are more capable to absorb and retain the dyes being applied *(Kramer, 2017).*
- It has a high heat absorption, allowing for adaptation in changing environmental conditions. Thermal comfort is a major characteristic of textiles comfort evaluation and can be gauged by the heat transfer mechanism. The thermal conductivity of the fibres, higher than the air one, and the capillary structure of fibres strongly influence the heat transfer phenomena *(Stanković et al., 2008)*. The porous nature of hemp allows the fabric to make it cool in warm weather. The same principle applies to cooler weather. The air trapped in the fibres of a garment is naturally warmed by the body keeping the warmth.
- It has anti-static properties, its strength does not collect electrostatic charges *(Kozłowski et al., 2020)*
- It has the ability to protect against UV radiation. Fabrics with at least 50% hemp content block the sun's UV rays more effectively than other fabrics *(Kozłowski et al., 2020).*
- It has a very high adsorbability to toxic gases, which is an advantage in household textiles *(Kozłowski et al., 2020).*
- It is naturally resistant to mould and has antimicrobial properties. The introduction of hemp fibres into a blend was reported to improve durability and hygienic properties of the fabrics *(Cierpucha et al., 1997).*
- Hemp fibres are strong and hold its shape in a fabric, stretching less than other natural fibres. This keeps the garment from stretching out or distort with use.
- Hemp fibres were cited in the '90s as a comfortable and cool alternative to man-made fibres due to their ecologically and environmentally friendly properties *(Cierpucha et al., 1997).* According to a recent study on US consumers, bast fibres including hemp fibres were perceived as modern fibre *(Ju et al., 2016).*

However, hemp textile has still some limitations concerning textile comfort. Hemp has low uniformity, is stiff and does not have much elasticity *(Stanković et al., 2019)*. In order to bring more comfort to the hemp, different investigations were carried out. For example, chemical treatments were found to be an effective way to improve the flexibility and softness of hemp woven fabrics *(Ji and Lee, 2016; Zhang and Zhang, 2010)*. Hemp blends with other types of fibres and fabrics design investigations were also found to have a positive effect on tactile comfort *(Novaković et al., 2020; Stankovic and Bizjak, 2014)*. Indeed, properties of textiles are mainly influenced by the design of two other structure levels, being the yarn and the fabric (Stanković et al., 2019).

1.1.2.2. Production of Hemp Nowadays

Today, hemp is mainly grown for seeds used as food additives for humans and animals, and is being explored for medicinal and therapeutic applications (Figure 1.7).

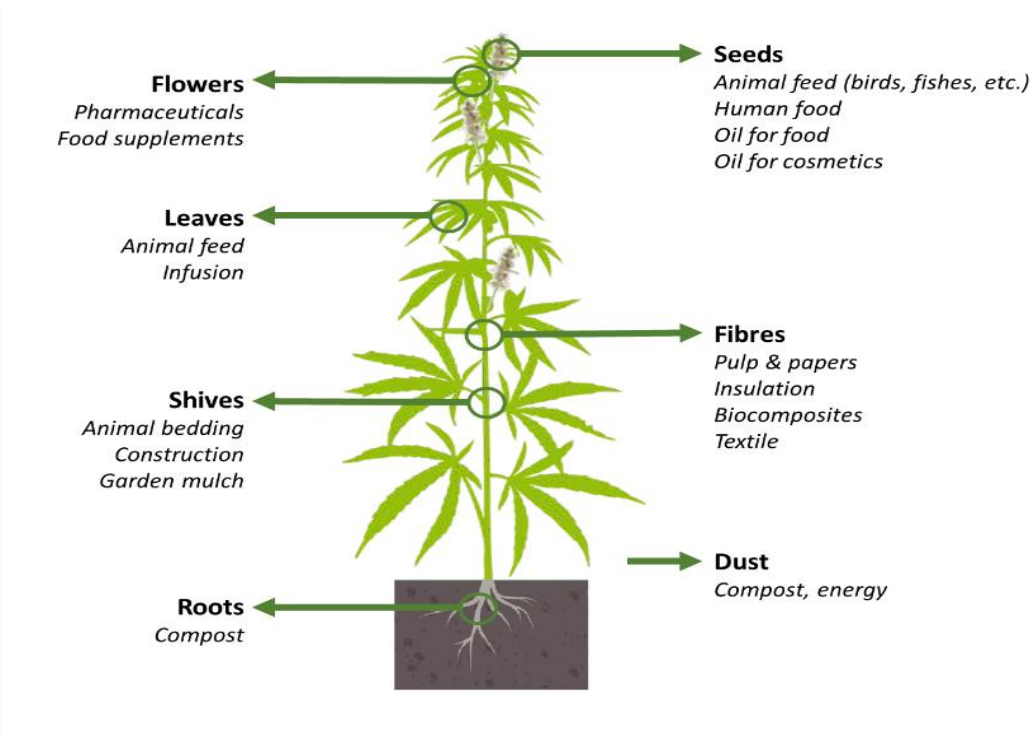


Figure 1.7: Uses of hemp, inspired from (Interchanvre, 2020).

Hemp is also cultivated for the short fibres used for building materials, papermaking, and composite materials *(Visković et al., 2023a)*. In addition, the development of industrial hemp for its long fibres for textile and composite applications is emerging. The growing demand for products with low environmental impact and local products encourages the development of reliable local production of textile fibres in Europe and worldwide (Ju et al., 2016; Zimniewska, 2022).

Hemp in the World

Nowadays, the major hemp growing countries are Canada, China, United State and France (Figure 1.8).

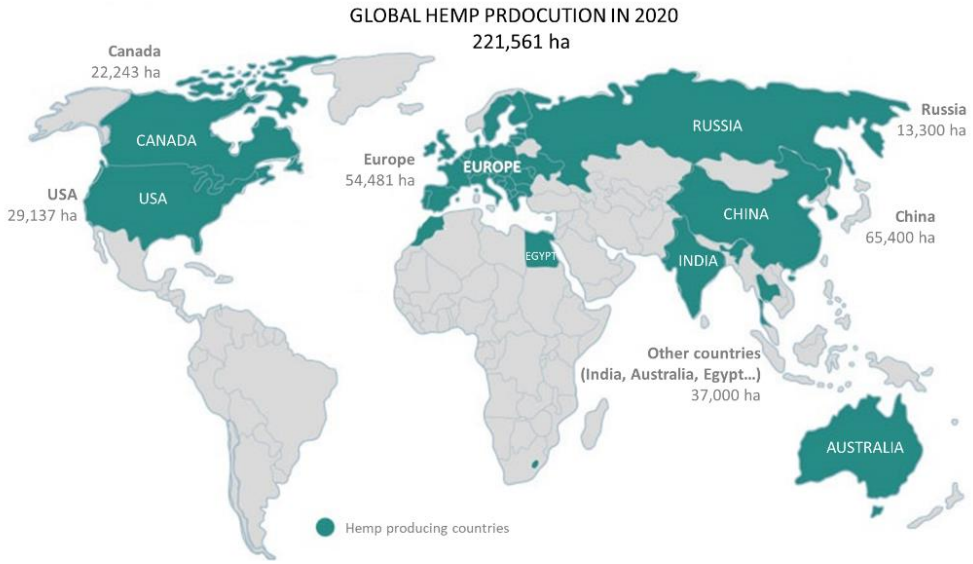


Figure 1.8: Hemp world production in 2020 (Interchanvre, 2022a).

Hemp in Europe

In recent years the area dedicated to hemp cultivation has increased significantly in Europe. The area of hemp cultivation in Europe increased by 70% from 2013 to 2018 (Figure 1.9).

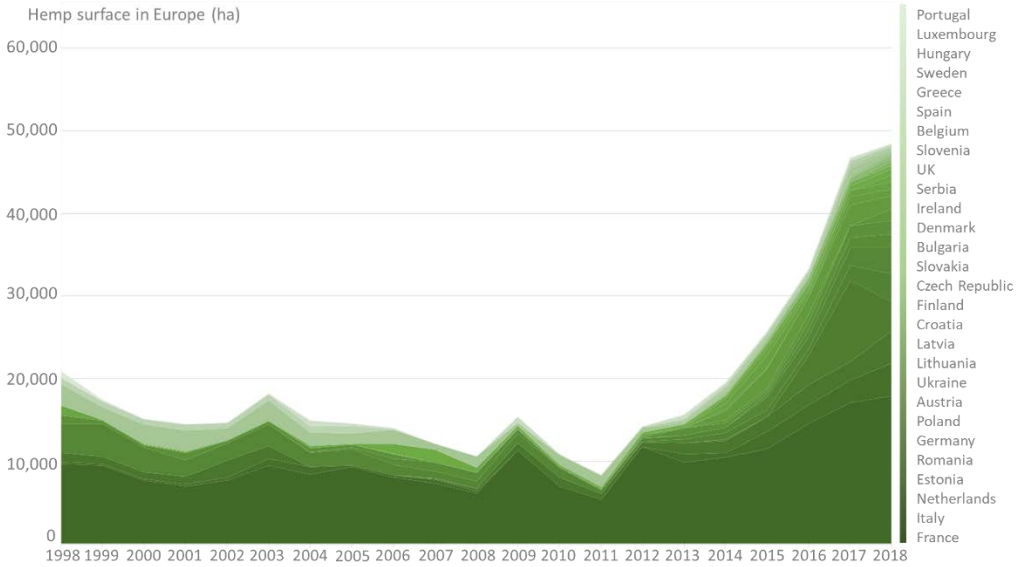


Figure 1.9: Evolution of hemp cultivated areas in Europe between 1998 and 2018 (EIHA, 2018).

In 2020, areas cultivated for hemp reached 54,481 hectares in Europe. France is the largest producer, accounting for more than 36% of EU production (Figure 1.10).



Figure 1.10: Distribution of hemp cultivated areas in Europe in 2020 (Interchanvre, 2022a).

Hemp in France

In 2022, area used for hemp cultivation reached 21,700 hectares in France, including 800 hectares for textile hemp (Figure 1.11). Seeds and straw represent respectively 21% and 79% of the economic value derived from cultivation.

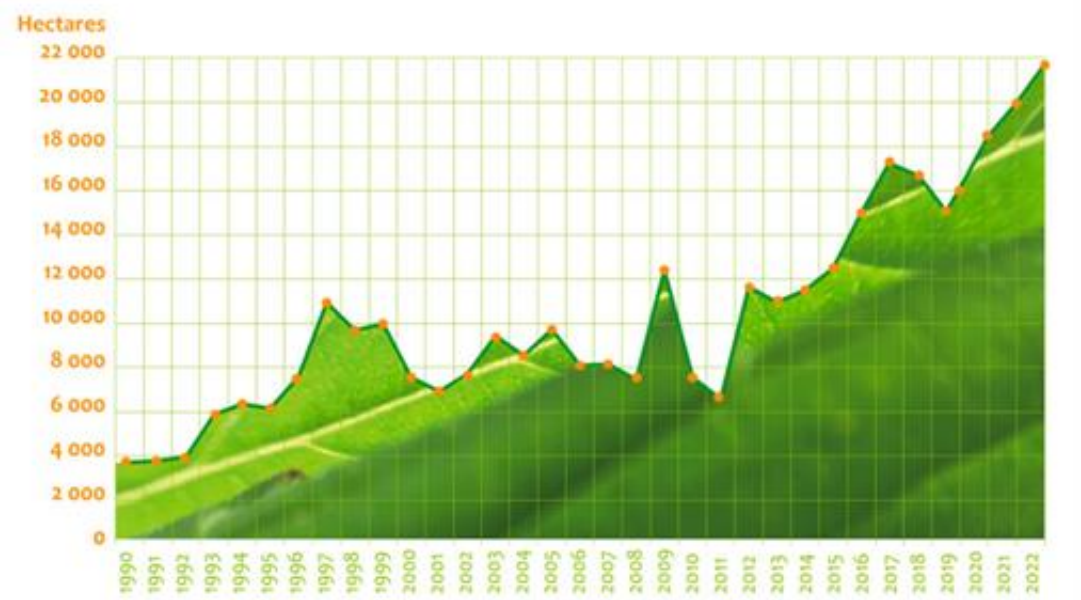


Figure 1.11: Evolution of hemp cultivation area in France (Interchanvre, 2022a).

1.1.3. The Hemp Plant

1.1.3.1. Growth Cycle of the Hemp Plant

The hemp plant belongs to the botanical family called Cannabaceae and the species Cannabis sativa L., which is commonly subdivided into three subspecies: Cannabis Sativa L. ssp. Sativa, Cannabis Sativa L. ssp. Ruderalis, Cannabis Sativa L. ssp. Indica *(Small, 2015; Small and Cronquist, 1976).* Textile hemp belongs to the Cannabis Sativa L. ssp Sativa subspecie. It is an annual dicotyledonous plant as it has two cotyledons and completes its life cycle within one short growing season. Life cycles last between 4 and 6 months but may be longer or shorter depending on several variables *(Hall et al., 2012)*. During its growth cycle, the plant goes through the germination and emergence stages, when the seed germinates, and the first leaves appear. Then the vegetative stage corresponds to the growth of the stem and the appearance of leaves, and the flowering stage when flowers appear. Seed maturity and then senescence takes place when the plant dries up (Figure 1.12) *(Institut Technique du Chanvre, 2007)*. Usually hemp stops growing soon after full flowering *(Mediavilla et al., 1998)*.

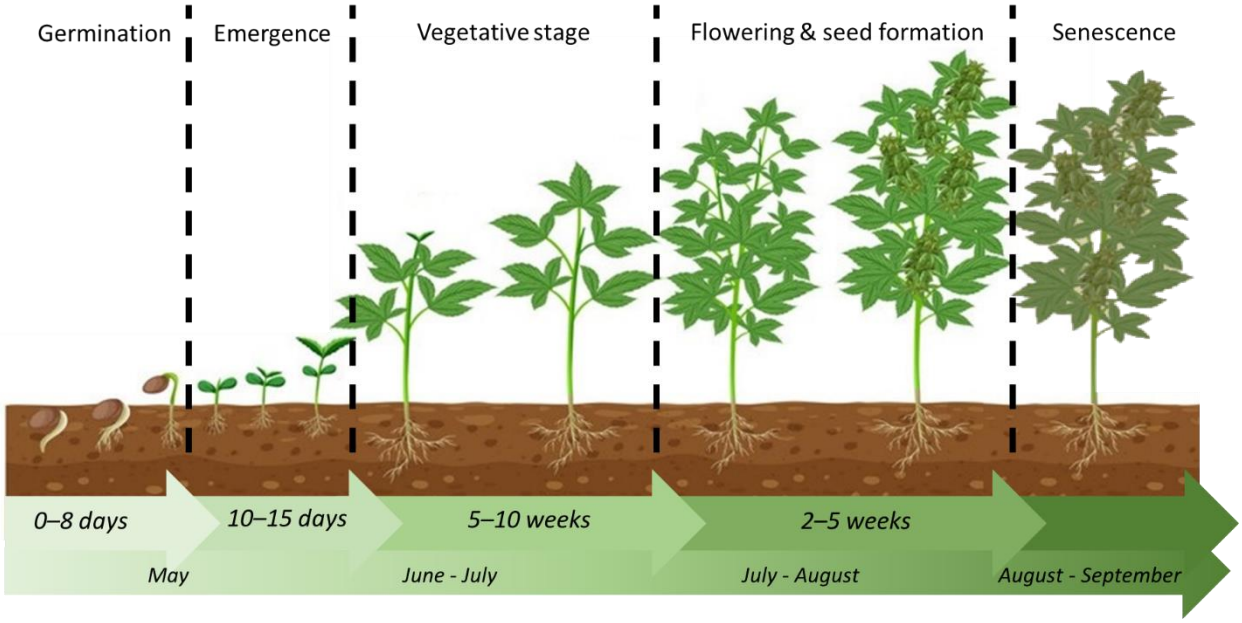


Figure 1.12: Growth cycle of hemp plant.

It naturally reaches a growth height ranging from 1 m to 4 m depending on species and pedo-climatic conditions *(Bonnétat, 1919).* The plant has a taproot and a branched root system which evolves in depth structuring the soil and fetching water from a wider area in case of water stress *(Bouloc, 2006)*. The stem is made up of internodes of varying length, resulting from differential growth. The leaves have lanceolate leaflets with unequal sizes. The leaves insert oppositely on the stem until the start of flowering, then the arrangement of the leaves (phyllotaxis) switches to alternating mode *(Bouloc, 2006).* In matured plants sown at a high density, the leaves from the lower stem part gradually become yellow and drop off *(Westerhuis, 2016).* The flowers form inflorescences are arranged in bunches at the top of the stem. The hemp seed is bivalve with an ovoid shape and typically contains 30%-40% oil and about 25% protein, with considerable amounts of dietary fibre, vitamins and minerals. It can be pressed to extract the oil, which is an exceptionally rich source of the two essential fatty acids: linoleic acid (omega-6) and alpha-linoleic acid (omega-3) in a ratio between 2:1 and 3:1, which is considered to be optimal for human health *(Callaway, 2004).*

1.1.3.2. Varietal Selection

Since the beginning of agriculture, crops have been domesticated by humans for their needs and plants with the most attractive characteristic were kept over the years. About 200 years ago, a new form of crop selection began with the crossing of plants by breeders, referred to as varietal selection *(Goudenhooft, 2018)*. For hemp plant, the major goal of varietal selection is to increase the plant yields, in terms of fibre content or seeds or woody core content depending on the end uses *(Thouminot, 2015)*. Characteristic such as the flowering system or the sexual dimorphism were proven to influence the plant yield and can be selected by breeders *(Faux et al., 2016)*. For textile hemp, specific requirements such as obtaining high bast fibre yields, the formation of fine and homogenous fibres, the limitation of lignified fibres or the adaptation to certain environmental conditions are challenging the breeders *(Shuvo, 2020)*. Recent form of breeding referred to as plant biotechnology involving DNA technology and marker aided selection are currently helping facing nowadays challenges *(Clarke and Merlin, 2016; Goudenhooft, 2018).* For example, extensive selection performed over many years on flax varieties led to an important increase in fibre yield by the improvement of the amount of fibres and the fibre bundles organisation *(Goudenhooft et al., 2017).*

A varietal development is a long process, including different steps *(Interchanvre, 2022b)* as presented below and in Figure 1.13:

- Identifying genetic resources (N=0)
- Characterisation of resources by phenotyping and genotyping
- Selection, creation of a new cultivar and indoor test (N+2/N+6)
- Field test with the selected varieties (N+7/N+8)
- Registration to the official catalogue of varieties
- Seed multiplication (N+11)
- Marketing (N+12)

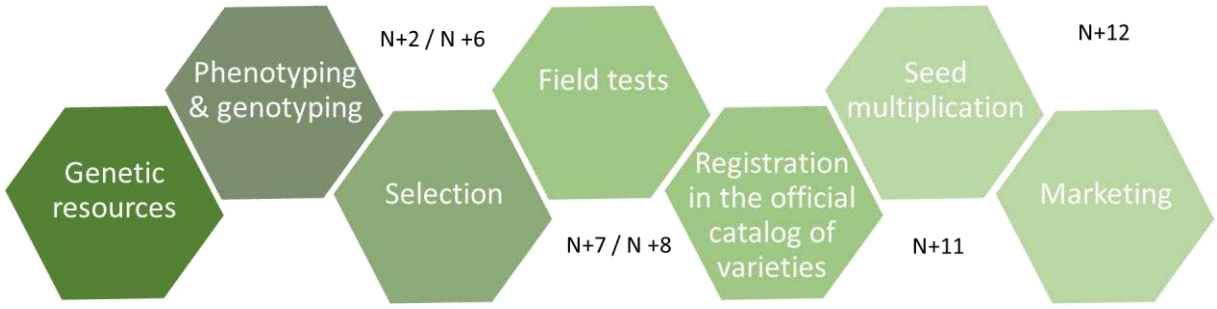


Figure 1.13: Scheme of varietal selection inspired from Hemp-it company.

The genotyping is the process of determining differences in the genetic makeup of an organism by examining their DNA sequence using various biological tests while the phenotyping is the determination of the set of observable characteristics or traits of an organism. For some simple traits, the correspondence between genotype and phenotype is direct, and the two sources of information are redundant. However, most qualitative traits depend on multiple genes, and the influence of the environment can be a determining factor. In this case, the genotype does not make it possible to precisely predict the phenotype of the individual, but only to estimate its average value. Selection and creation of a new cultivar is a long step by step task as it includes the indoor growth of the plants, the study of the genetic stability of the cultivars and the post-harvesting analysis such as fibres or seed yields depending on the objectives *(Petit et al., 2019; Salentijn et al., 2019)*. Out of a hundred varieties created, about ten are selected for field trials. Every year, the Hemp-it company (French seed

breeding company, Hemp-it, Beaufort-en-Anjou, France) add about 1 to 2 new varieties to the seed catalogue.

Moreover, a hemp-specific criterion in varietal selection is related to legal restrictions: the level of the psychoactive substance delta-9-tetrahydrocannabinol (THC) in the plant must be monitored to ensure it is lower than the legal limit. The legal limit is evolving over the years and with the latest research on human effects. From 2022, the EU has set the limit for THC content in hemp seed at 0.3% *(European Comission, 2023)*. This latter parameter is challenging breeders, as cultivars exceeding the legal THC restrictions are excluded from the European Common Catalogue. Likewise, raw hemp (stems, leaves, flowers) must have a THC content not exceeding 0.3% and farmers must use certified seed of varieties listed in the EU common catalogue of varieties which registers 75 different hemp varieties *(European Comission, 2023).*

1.1.3.3. The Monoecious and Dioecious Hemp Plant

The hemp plant is naturally dioecious, meaning that male and female flowers are borne on separate plants as presented in the Figure 1.14-A/B. Naturally, male and female plants occur in a well-balanced ratio of 1:1 *(Mediavilla et al., 1998; Schumann et al., 1999).*

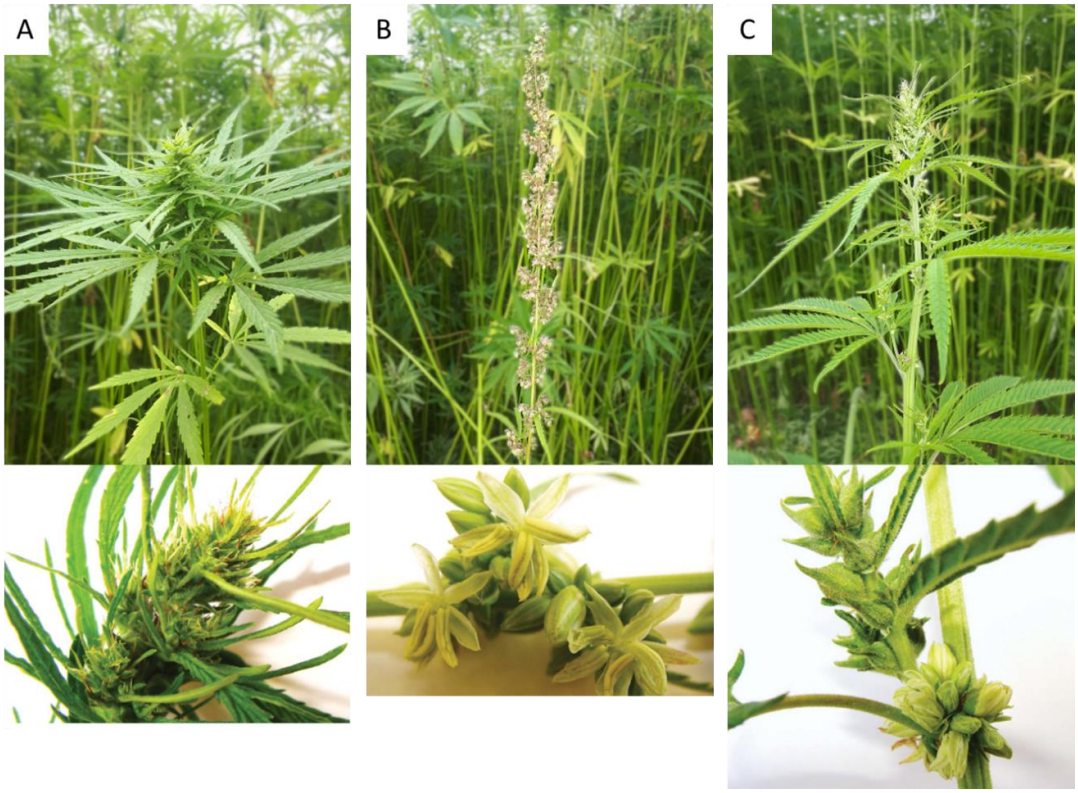


Figure 1.14: A female flower (A) and a male flower (B) from a dioecious hemp variety and flower (C) from monoecious hemp variety. Enlargement from (Hall et al., 2012).

Gender difference is mainly identifiable at the period of inflorescences, as males have a branched inflorescence in the top of the plant with a little number of leaves whereas females have its inflorescence at the apex of the stem *(Bouloc, 2006)*. Moreover, flowers tend to appear earlier on male plants, about two weeks before the ones on female plants *(Mediavilla et al., 1998; Struik et al., 2000).* This shift is specific to each variety. The genders are dimorphic not only as regards to the reproductive organs but also on the plants morphology. However prior to the formation of reproduction structures, the two sexes are not visually distinguishable *(Campbell et al., 2021; Hall et al., 2012; Small, 2015).* Male plants tend to grow a little taller

than female plants by up to 15% in average at maturity, even though it is varying with varieties. They form less and smaller leaves and thinner stems and die after shedding their pollen *(Small, 2015).*

For textile applications, the fibre quality of male plants was considered superior to the fibre quality of female plants. They are also reported to have a higher fibre percentage in mass. In the past, hemp harvesting was carried out by hand and the male and female plants were collected separately (Bonnétat, 1919; Lévêque, 1925). Male plants were often harvested earlier than the female plants, which had to wait until seed ripeness. By the time of seed ripeness, the male plants could be deteriorated with a simultaneous harvesting. The fibres from male plants, thin and soft, were intended for the finer fabrics, whereas fibres from female plants, thick and strong, were used for rougher textiles or ropes, as explained in the Er Ya dictionary *(Chinese dictionary 200 BC) (Bonnétat, 1919; Lévêque, 1925; Lu and Clarke, 2012).*

The chemical composition has been reported to diverge between male and female plants. Talley (1934) conceded that high carbohydrate levels in male plants and higher N levels in females are related to the fact that flowering exhausts male plants that are no longer capable of N assimilation from the soil while females continue to acquire nutrients during grain filling *(Hall et al, 2012).*

For reasons of homogeneity, industrialisation and ease of harvesting, genetic selection work was carried out to obtain monoecious varieties, meaning that the male and female flowers are borne by the same plant *(Westerhuis, 2016).* Monoecious plants can occur naturally, and breeders used the natural monoecious plant for breeding programs and development of new varieties. The female flowers develop at the apical part of the plant while the male inflorescences are distributed on the stem at the alternate nodes as presented in Figure 1.14-C. Monoecious is considered appropriate for a dual-purpose application to harvest both seed and fibres *(Baldini et al., 2018)*. It leads to a higher seed yield but smaller and less vigorous pollen grains, a higher crop homogeneity than dioecious plants and an easier mechanical harvest due to synchronised maturity and morphology *(Small, 2015)*. However, their sexual phenotype is unstable. Those artificial varieties tend to return to the dioecy so their seeds multiplication requires a strict elimination of the occasionally occurring dioecious male plants *(Amaducci et al., 2008b)*.

1.1.3.4. The Photoperiodicity

Hemp is a photoperiodic plant. Photoperiodicity reflects the influence of the length of day and night on various physiological reactions in the plants *(Tournois, 1912).* Short-day plants require a minimum dark period or “short day” to trigger the transition from the vegetative to reproductive phase *(Hall et al., 2012)*. Flowering is induced under a certain critical day length; short days accelerate flowering *(Lisson and Mendham, 2000)*. The cultivars grown in Europe (in the beginning of the 21st century) have generally a photoperiod between 12 h to 15.5 h *(Struik et al., 2000).*

The flowering behaviour is depending on temperature, light quality and the length of the photoperiod *(Amaducci et al., 2008a; Salentijn et al., 2019)*. The photoperiodicity is directly linked with the latitude of growth: plants grown in the northern latitudes tend to produce a longer vegetative phase, while at lower latitudes plants tend to flower faster due to shorter daylight.

The sensitivity to the photoperiod is very different depending on the varieties. Generally, hemp plants are defined by their precocity. It corresponds to the full flowering stage which is the time when 85% of the (female) plants have reached the stage of opening of the

last female flowers on the apical part (Interchanvre, 2017). Based on flowering time, three main groups may be distinguished: early (flowering after 40-60 days), intermediate (60-90 days) and late (90-120 days) varieties *(Amaducci et al., 2008b; Zatta et al., 2012)*. At Paris (France) latitude, early/intermediate-flowering varieties reach full flowering by July while late-flowering varieties reach full flowering after mid-August *(Hemp-it company).*

Thus, the early-flowering varieties are generally favoured at high latitude for harvesting seeds. It allows time for seeds to mature before the onset of a killing frost and very humid weather, while the late-flowering varieties could be favoured at high-latitude to obtain fibres in quantity (Interchanvre, 2017; Salentijn et al., 2015). The later the variety, the higher and larger the stems and the greater its potential stem yield (Hall et al., 2014).

1.2. Structure of Hemp Fibres

1.2.1. Structure of Bast Fibres

1.2.1.1. Fibres Inside the Stem

The hemp stem is arranged, from the inside to the outside, of an empty pith, xylem, phloem, and bark as presented in Figure 1.15.

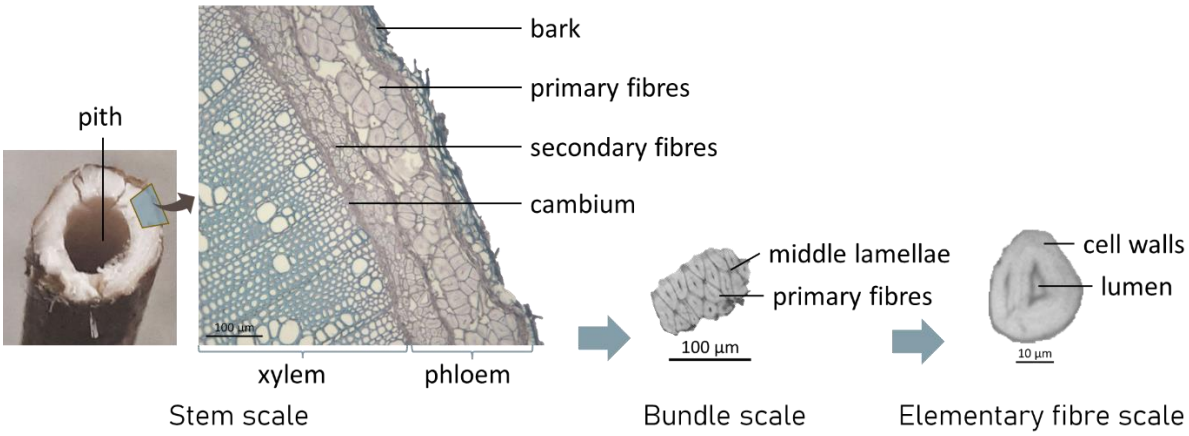


Figure 1.15: Representation of the structure of a hemp stem (inspired from (Charlet, 2008)).

The xylem is rich in lignin, radially organized, and carries raw sap. Raw sap consists in water with mineral elements, hormones and some nutrients and is moving from the roots toward the leaves. The phloem transports the elaborated sap. The elaborated sap consists mainly in sugars, hormones and mineral elements dissolved in water and flows from the leaves toward the whole plants. The bark, also called the epidermis, protects the stem. Located between the xylem and the phloem, the cambium is at the origin of the generation of a secondary system *(Aloni, 1987)*. The phloem is made up of parenchyma cells, which serve, among other things, as a reserve or as a filling, and sclerenchyma cells, such as bast fibres with a supporting role. The adjacent fibres are glued together by a pectin-rich intercellular material named middle lamellae. They can be grouped in the form of bundles gathering in average thirty fibres *(Bourmaud et al., 2018).*

1.2.1.2. Fibre Structure and Layers

Botanically, the fibres consist of an individual cell characterised by a thick cell wall of polygonal shape and a significant length with a length-to-diameter ratio higher than 1,000 *(Snegireva et al., 2015; van Dam and Gorshkova, 2003).* Elementary hemp bast fibres have a

length of the order of few centimetres; the average length is between 35 mm and 40 mm but it can vary from 5 mm to 100 mm, and a diameter of a few tens of microns; about 10 µm to 50 µm *(Gorshkova et al., 2012; Sponner et al., 2005).* The main constituents of the fibres are cellulose, hemicellulose, lignin, pectin, waxes and fats *(Sponner et al., 2005)*. Bast fibres are made of a hollow area in the middle, called lumen, which contributes to water circulation and its size is an indication of the maturity of the fibre. Small lumen size generally indicates a good fibre maturity. Surrounding the lumen, the cell walls are divided in two categories: an external wall called primary cell wall and an internal one called secondary cell wall, as presented in the Figure 1.16.

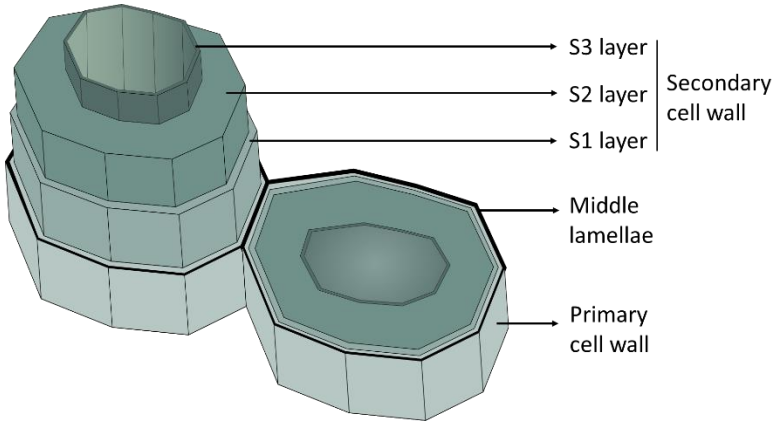


Figure 1.16: Scheme of cell walls of hemp bast fibres.

The primary cell wall has a thickness in the range of one tenth of a micrometre (for flax it varies between 0.1-0.2 µm) and it is composed of cellulose microfibrils embedded in a matrix with amorphous pectin, hemicellulose and lignin *(Gorshkova et al., 2010; Melelli et al., 2020).* It has a chemical composition close to that of the middle lamella *(Gorshkova et al., 2010; McDougall et al., 1993; Rezvani Moghaddam and Wilman, 1998; Zamil and Geitmann, 2017).* The secondary cell wall is the thickest wall since it represents up to 80% of the mass of an elementary fibre at maturity. It is weakly lignified, rich in cellulose and is mainly responsible for the mechanical properties of the fibre *(Bourmaud et al., 2018; Chernova and Gorshkova, 2007; Gorshkova et al., 2010; Kiyoto et al., 2018).* Its cell wall thickness reaches 5 µm to 14 µm in mature plants *(T. Chernova et al., 2018; Crônier et al., 2005).* Its internal organisation is discussed by numerous authors; however, it is generally, as defined by Roelofsen (1951, 1959), consisting of spiral structure with three distinct layers: S1, S2 and S3, going from the outside to the inside of the fibre *(Roland et al., 1995).* The classification is mainly based on the thickness, the microfibril orientation and the chemical composition *(Clair et al., 2018).*

1.2.1.3. Secondary Cell Wall and Microfibrillar Angle

The three layers of the secondary cell wall are made up of concentric strata of cellulose microfibrils in a matrix of pectin, lignin and hemicellulose. Cellulose microfibrils are organized parallel to each other and in a spiral winding with S or Z-helix orientation *(Roelofsen, 1966).* They are a linear association of crystalline cellulose chains. They measure several microns in length and few nanometres in diameter (10 nm to 30 nm) *(Zimniewska, 2022).* The microfibrils containing 30-100 cellulose molecules in the chain give mechanical strength to the fibres *(Zimniewska, 2022).* They are grouped into macrofibrils of several hundred nanometres wide *(Baley, 2002)*.

The angle of the cellulose microfibrils to the longitudinal axis of the fibre is called the microfibrillar angle. The orientation of the cellulose microfibrils can be determined by Atomic

Force Microscopy (AFM) *(Bourmaud et al., 2018)*, by X-ray Diffraction (XRD) at both small (SAXS) and wide angles (WAXS) *(Cave, 1997; C. Wang et al., 2018)*, by Second Harmonic Generation (SHG) *(Melelli et al., 2020)* or by polarised Raman microspectroscopy *(Thygesen and Gierlinger, 2013).* The microfibrillar angle varies from layer to layer of the *fibre (Gorshkova et al., 2010).*

The outer layer S1 has cellulose microfibrils transversely oriented with a high angle varying between 70° and 90° *(Dai and Fan, 2010; Thygesen et al., 2006).* The microfibrillar angle of the S2 layer is the smallest with values close to 1-10°, meaning that the orientation is close to the fibre axis, and has Z-helical orientation, which means right-handed fibre *(Bergfjord and Holst, 2010; Bledzki and Gassan, 1999a; Dai and Fan, 2010; Placet et al., 2011; Thygesen et al., 2005).* The microfibrillar angle as well as the level of crystallinity of the cellulose constituting the microfibrils are key parameters to describe the tensile properties of a fibre. The structure of this layer is primarily responsible for the mechanical properties of the secondary cell wall and so the fibre. Lastly, the orientation of the microfibrils in the inner S3 layer was found almost parallel to the longitudinal axis *(Haugan and Holst, 2013).* The size of the different layer of the cell wall of flax fibres are presented in Table 1.1.

Table 1.1: Dimension of cell wall layers in flax fibres (Baley et al., 2005; Bourmaud et al., 2013; Melelli et al., 2020; Nilsson and Gustafsson, 2007; C. Wang et al., 2018).

Primary cell wall	0.1-0.2 µm
Secondary cell wall – S1	0.2-5 µm
Secondary cell wall – S2	5-10 µm
Secondary cell wall – S3	0.5-1 µm

Hemp fibres and flax fibres show many similarities in terms of structure, layers organisation, biochemical composition *(Bourmaud et al., 2018)*. However, some differences can be highlighted *(Del Masto et al., 2019)*. Hemp fibres tend to have smaller fibres in length with a bigger diameter than flax fibres *(Bergfjord and Holst, 2010; Charlet et al., 2010; Müssig, 2010; Thygesen et al., 2007).* The shape of the cross-section is generally closer to a perfect circle for flax fibres than for hemp *(Gregoire et al., 2023)*. The microfibrillar orientation was observed to be S-twist for flax and Z-twist for hemp with a similar range of microfibrillar angle *(Bergfjord and Holst, 2010; Dai and Fan, 2010)*. Flax shows a higher percentage of crystalline cellulose *(Bourmaud et al., 2018).* Flax fibres exhibit higher values of tensile properties than hemp especially in terms of tensile strength, stiffness and deformation *(Grégoire et al., 2021).* However, values of elastic modulus of their cell wall by peak-force nano-mechanical properties are similar or even higher for hemp *(Bourmaud et al., 2017; Goudenhooft et al., 2018).* Mechanical, morphological and biochemical values are generally more scattered for hemp than for flax, characterising a more heterogenous fibre.

1.2.1.4. Mechanical Properties and Defects

The mechanical properties of fibres are dependent on their microstructure and their composition. Cellulose is the main chemical component and it is responsible for the mechanical properties of plant fibres *(Bourmaud et al., 2018).* The rate of crystallinity of cellulose has an influence on the stiffness of the fibres; hemp or flax fibres with high levels of crystalline cellulose exhibit high properties *(Richely et al., 2022).*

Mechanical properties of the fibres are most of time determined by tensile test. In general, plant fibres do not exhibit all the time a perfect linear elastic behaviour during tensile test. Three types of stress-strain curve are observed, as drawn in Figure 1.17: type 1 is a linear curve typical of a truly elastic behaviour; type 2 has a non-linear curve with a decrease in

stiffness above the elastic limit describing a plastic flow, and type 3 exhibits a non-linear region in the earlier stage of the loading and a linear region in the second stage and has commonly been described as characteristic of natural fibres *(Duval et al., 2011; Pickering et al., 2007)*.

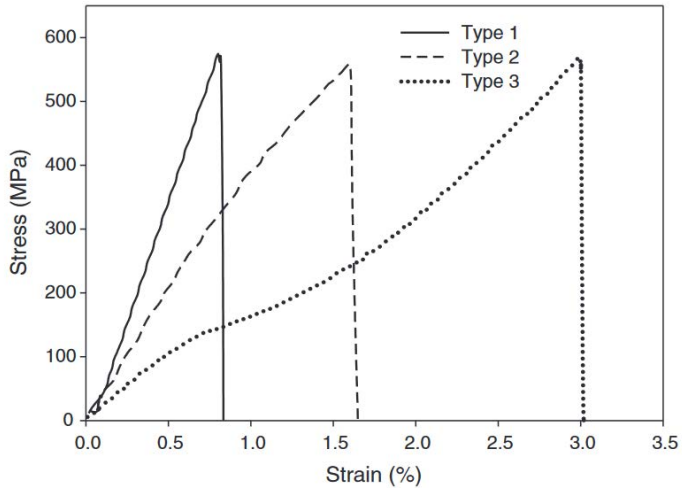


Figure 1.17: Typical stress-strain curves of bast fibres from (Duval et al. 2011).

Tensile properties of hemp fibres (single or bundles) found in literature are presented in Table 1.2.

Table 1.2: Tensile properties of hemp fibres from literature.

Author	Young's modulus (GPa)	Strength at break (MPa)	Strain (elongation) at break (%)
(Sankari, 2000)	-	830	4.3
(Thygesen et al., 2006)	24.9 ± 10.6	1735 ± 723	-
(Pickering et al., 2007)	58	857 ± 260	-
(Bourmaud and Baley, 2009)	44.5 ± 19.1	788 ± 307	1.8 ± 0.69
(Fan et al., 2011)	66	886	-
(Ringuette, 2011)	70	550 - 900	1.6
(Duval et al., 2011)	19.6 ± 14.8	482 ± 337	3 ± 1.5
(Sawpan et al., 2011)	26.5	577	-
(Placet et al., 2012b)	24.7 ± 11.4	636 ± 253	2.1 ± 0.7
(Marrot et al., 2013)	31.2 ± 19.7	699 ± 450	3.3 ± 1.6
	35.5 ± 17.3	889 ± 472	2.6 ± 2.2
(Liu et al., 2015a, 2015b)	34.9 ± 4.4	954 ± 162	6.2 ± 1.2
	26.4 ± 5.9	686 ± 126	4.66 ± 1.31
(Placet et al., 2017)	16.7 ± 6.7	345 ± 200	3.07 ± 1.1
(Bourmaud et al., 2018)	14.4 - 44.5	285 - 889	0.8 - 3.3
(Réquilé et al., 2018)	24.22 ± 16.64	406.7 ± 255.7	2.87 ± 1.65
(Mazian et al., 2019)	17	487	3.4
(Grégoire et al., 2021)	49 ± 19	875 ± 367	-

It is important to note that tensile tests are carried out with different protocols and either on bundles of fibres or on elementary fibres, which do not lead to the same conclusions.

The sampling area, the shape of the cross-section and the humidity level were observed to have an impact on the type of stress-strain behaviour obtained and the mechanical performance of the fibres *(Baley et al., 2005; Fan, 2010; Garat et al., 2020, 2018; Gregoire et al., 2023)*. The approximation of the measured cross-section area for the tensile calculation highly influences the results and in this way it was shown that an elliptical model gives more accurate results than a circular model *(Garat et al., 2018; Gogoli et al., 2021; Gregoire et al., 2023).* It has been observed in flax plants that the fibres from the central part of the stem possess the best mechanical properties *(Charlet, 2008).* Similarly, the size of the lumen, and therefore the level of maturity of the fibres have an impact on the mechanical properties of the fibres *(Fuentes et al., 2019; Placet et al., 2014b)*. However, some studies have shown that fibres collected at the end of flowering exhibit higher tensile strength, tensile moduli and strain at break than those collected at seed maturity *(Liu et al., 2015a).*

The presence of structural defects in the fibres can affect the mechanical properties *(Richely et al., 2022).* Dislocations named kink-bands are the most common defects inside plant fibres. They result from a discontinuity in the crystalline organisation and a variation of the micro fibrillar structure. The microfibrillar angle in the dislocations reaches approximately 30° and a transition zone surrounding the large dislocations has an intermediate orientation with of microfibrillar angle of approximately 10-15° *(Thygesen and Gierlinger, 2013).* They are distributed heterogeneously over the entire length of the fibre *(Bourmaud et al., 2018).* At the surface of the fibres, disordered patterns are revealing the kink-bands and can be observed by bright-light microscopy or scanning electronic microscopy (SEM) imaging (Figure 1.18) *(Baley, 2004; Melelli et al., 2020; Thygesen et al., 2006).* Inside the transition zones, the presence of pores is revealed and can be observed by SEM, atomic force microscopy (AFM) and micro-tomography imaging (Figure 1.19) *(Abbey et al., 2010; Bos and Donald, 1999; Bourmaud et al., 2022; Melelli et al., 2021; Quereilhac et al., 2023; H. Zhang et al., 2015).* Moreover, the change in the crystalline organisation can be observed by second harmonic generation (SHG), polarised light microscopy (PLM) or polarised Raman microspectroscopy *(Melelli et al., 2021; Thygesen and Gierlinger, 2013).* The cell walls of these areas have a greater microfibrillar angle than the rest of the fibre *(Melelli et al., 2020).* It was reported that more defects were visible with PLM than SEM and it was suggested that SEM allows observing only the largest dislocations *(Thygesen et al., 2006).* The defects are favourable areas for the initiation of a crack and they seem to reduce the stiffness of the fibre *(Beaugrand et al., 2017; Guessasma and Beaugrand, 2019; Nilsson and Gustafsson, 2007; Richely et al., 2021; Thygesen et al., 2007).* These defects are a privileged point of entry for water, micro-organisms or enzymes inducing a more rapid alteration and degradation of the fibres *(Foulk et al., 2008; Hernandez-Estrada et al., 2016; Thygesen et al., 2011).*



Figure 1.18: Kink-bands observed by SEM micrographs (left) and under polarised light (right) (Melelli et al. 2021) (Grégoire 2020).

Dislocations are reported to be created when the fibre wall is subjected to uniaxial compression in the direction of the fibres or bending while still being in organized bundles *(Nyholm et al., 2001; Terziev et al., 2005)*. The apparition of defects is not well defined, they can appear during the growth of the fibres and bundles or when they are subjected to environmental stresses *(Richely et al., 2022).* However, the number and the severity of the defects are reported to increase during the postharvest treatments, such as scutching and hackling processes or decortication process *(Bos et al., 2002; Hänninen et al., 2012; Hernandez-Estrada et al., 2016).* Interestingly, cotton fibres which are not load-bearing in the plant, do not seem to contain dislocations in the native state *(Thygesen and Gierlinger, 2013).*

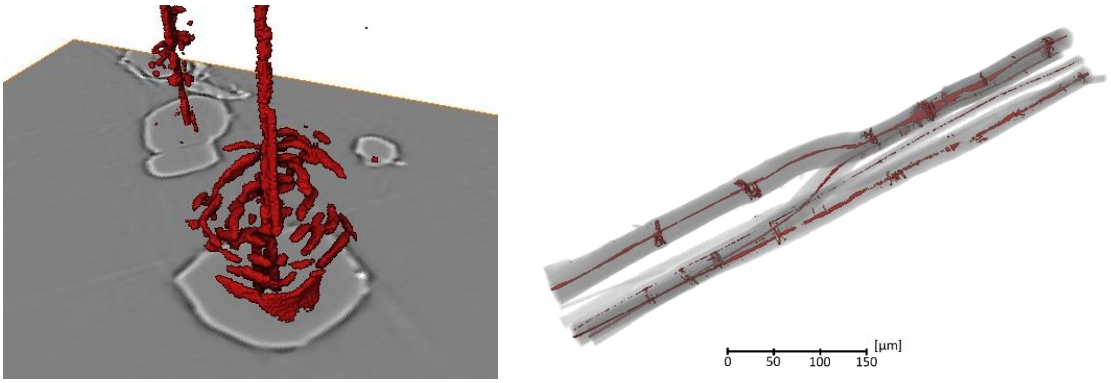


Figure 1.19: Micro-tomography images of dislocation areas inside flax fibres. Red colour represents pores. (Bourmaud et al., 2022).

1.2.2. Chemical Composition

The chemical composition of hemp fibres, reported from the literature, is summarised in Table 1.3. The percentage values of the main chemical components of hemp fibres given by different authors differ substantially from each other. The chemical composition can be determined by chemical analysis *(Bredemann, 1942; Crônier et al., 2005; Toonen et al., 2004*), infrared spectroscopy *(Fernandez-Tendero et al., 2017)*, Raman *spectroscopy (Coste et al., 2020)*, immunohistochemistry *(T. E. Chernova et al., 2018).* The distribution of chemical components in the fibre depends on several aspects, such as hemp plant variety, climatic conditions of growing, level of maturity, retting stage, method of fibre extraction. All these parameters explain the large differences between the values found in the literature. Hemp fibres consist mainly in cellulose, hemicellulose, lignin and pectin in different proportions. The rest consists in mainly minerals, waxes and water-soluble components *(Carpita, 1990).*

Table 1.3: Biochemical composition of hemp fibres from literature.

Author	Cellulose	Hemi-cellulose	Lignin	Pectin	Fat, Wax and ashes
(Bismarck et al., 2002)	67	16	3.3	0.8	2.8
(van Dam and Gorshkova, 2003)	70	15	3	3	/
(Wang et al., 2003)	67-78.3	5.5-16.1	2.9-3.3	0.8-2.5	
(Baltazar-y-Jimenez and Bismarck, 2007)	75	2	8		3.5
(Kostic et al., 2008)	76	12	5.7		
(Marrot et al., 2013)	80	12	2.6	3.0	2.2
(Angelini et al., 2016)	66	6.9	2.5		
(Bourmaud et al., 2018)	55-90	12	2-5	3	1.7
(Manaia et al., 2019)	70-74	15-20	3.5-3.7	0.5-1	2.0-7.0
(Lyu et al., 2021)	55-77	14-22.4	3.7-13	0.9	2

1.2.2.1. Cellulose

Cellulose is the main component of plants. The repeating unit in the cellulose chain is strictly speaking cellobiose, consisting of two glucose units as shown in Figure 1.20.

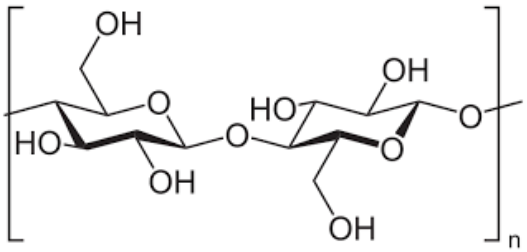


Figure 1.20: Structure of cellulose.

It is a polysaccharide consisting of linear chains of D-glucose molecules linked together by β-(1-4) bonds. It has three hydroxyl groups, secondary -OH at the C-2, secondary OH at the C-3, and primary OH at the C-6 position, which can form inter or intramolecular hydrogen bonds. These hydrogen bonds and Van der Waals forces promote the highly ordered organisation of cellulose molecules aligned together and give a fibrillar structure to the cellulose. The microfibrils can be more or less compacted thanks to hydrogen bonds to form crystalline or amorphous cellulose. The rate of crystallinity of cellulose in hemp fibres is dependent on the stage of maturity of the fibres and it has been measured up to 92% according to X-ray diffraction measurement *(Mwaikambo and Ansell, 2002; Pickering et al., 2007; Thygesen et al., 2007).*

Cellulose is hydrophilic, relatively resistant to oxidising agents, strong alkali treatment but can be hydrolysed by acid to water-soluble sugars. The degree of polymerisation is the key factor to characterise the supramolecular structure of cellulose. The degree of polymerisation (n or DP) is the number of glucose units in the chain. In general, increasing degree of

polymerisation correlates with higher mechanical strength. The degree of polymerisation is typically 2000-4000 glucose units to as high as 10,000–15,000 in the secondary cell wall. The degree of polymerisation was found to be 4400 in cotton, 8000 in flax and 2100 in pine pulp *(Abot, 2010; Bledzki and Gassan, 1999b; Evans and Wallis, 1989; Thygesen, 2006).*

1.2.2.2. Hemicellulose

The hemicelluloses are heterogeneous amorphous polymers. They are non-linear and highly branched polymers, on the contrary to cellulose, and mainly responsible for water absorption. They are made up of neutral monosaccharides (mainly xylose, mannose, galactose, glucose) and acids (glucuronic acids…). The skeleton is mainly consisting of xylan and xyloglucans and also contains mannans and mixed likage β-glucans. This diversity of chemical constituents makes hemicelluloses, in their native state, of amorphous nature, non-crystallisable and of low molecular weight. The degree of polymerisation is much lower than in cellulose ranging from 20 to 300 *(Sauvageon, 2019; Thygesen, 2006).*

Their hydrolysis is therefore relatively easier than that of cellulose. The hemicelluloses can be removed from the hemp fibre by extraction with aqueous alkali. The hemicellulose is embedded in the cell walls of plants and interacts with cellulose by creating links with cellulose. It forms hydrogen bonds with cellulose and covalent bonds with lignin.

1.2.2.3. Pectin

Pectin is a polysaccharide composed mainly of galacturonic acid (Figure 1.21) which linked by α-(1-4)-glycosidic bonds forms the main chain, the homogalacturonan chain (HG).

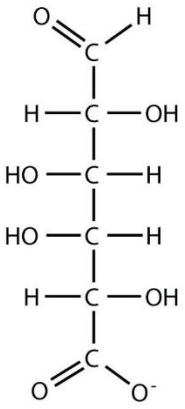


Figure 1.21: Representation of D-galacturonic acid in Fisher projection.

Other monosaccharides are also present such as rhamnose, galactose, arabinose or xylose. According to their distribution, two other main chains are formed: rhamnogalacturonan I (RG I) and rhamnogalacturonan II (RG II), both consisting of α-(1-4)-D-galacturonic acid and α-(1-2)-L-rhamnose units with different types of side branches. HG represents 70% of the pectin mass, RG I and RG II, 20-35% and 5-10% respectively *(Liu et al., 2016).*

The main chain of the polymer presents carboxylic acid functions that can undergo methoxylation. The degree of methoxylation (LM, low-methoxylated when more or less than 50% of carboxylic functions are esterified; or HM, high-methoxylated) corresponds to the proportion of methylated acid functions and has an impact on the physicochemical properties of pectin. HM pectin favours hydrogen bonding and forms a gel under acidic conditions in the presence of neutral sugars. LM pectin creates coordination bonds in the presence of divalent

cations (particularly Ca^{2+}), giving rise to a network and forming gels, as presented in Figure 1.22.

With the presence of calcium cations, the “egg-box” model is described, in which ionic bridges are formed between calcium ions and the ionised carboxyl groups of the galacturonic acid *(Mahé, 2018).* This formation allows to store a large amount of water.

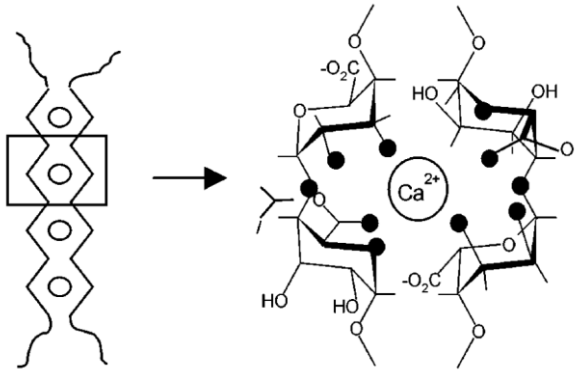


Figure 1.22: The “egg box” model representation, from (Mahé, 2018)

Pectic substances are the major constituents of the middle lamellae and ensure the cohesion of the bundle. They also play a role in the growth of the plants. They are present in both primary and secondary cell wall *(Behr et al., 2019; T. E. Chernova et al., 2018)* and are associated with the cell wall formation. They are largely present in the form of calcium pectate in primary cell wall. The linkages of the pectic components are mainly covalent in the inner part of the wall and are mainly by calcium bridges in the outer part which play the major part in intercellular adhesion *(Jauneau et al., 1992).*

Pectins are largely soluble in hot water. However LM pectins needs chelating agent or stronger treatment to be extracted *(Sauvageon, 2019).* Pectin can be depolymerised and extracted by moderate heating in acid or low acid medium, microwaves, ultrasounds, enzymes such as pectinase, chelating agent such as EDTA *(Mahé, 2018).* Retting preferentially reduces rhamnogalaturonic pectins than homogalaturonic pectins *(Sharma and Sumere, 1992; Vignon and Garcia-Jaldon, 1996).*

1.2.2.4. Lignin

Lignin is a phenolic polymer which complex three-dimensional structure is not well understood due to the lack of regular and ordered repeating units. The basic unit of lignin is the phenyl-propane type (C6-C3), and is classified among polyphenols. It is made up of a large group of organic polymers formed by cross-linking phenolic precursors. It is derived primarily from three types of monolignols giving rise to monomers which differ in their degree of methoxylation of their aromatic rings: paracoumaryl alcohol, coniferyl alcohol and sinapyl alcohol as presented in Figure 1.23.

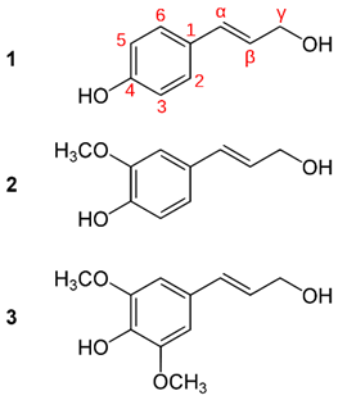


Figure 1.23: The three common monolignols: paracoumaryl (1), coniferyl alcohol (2), and sinapyl alcohol (3).

A single and precise definition of lignin is not definite because of its great variability. It is a three-dimensional polymer, without regular and ordered repeating units. It is hydrophobic as it is rich in aromatic units.

Lignin occurs in the spaces between cellulose, hemicellulose and pectin components in the cell wall. The key role of lignin in cell walls is supporting the structure of the plant tissue and ensures the transport of water and aqueous nutrients throughout the plant (Wang et al., 2003). While the majority of plant components are hydrophilic, lignin is hydrophobic as it is rich in aromatic units, and allows the plant vascular tissue to conduct water efficiently. The lignin forms a barrier for evaporation and, thus, helps to channel water to critical areas of the plant. In addition, it is a natural binding agent to fibrous lignocellulosic plant that protects the polysaccharides from destruction by fungi and bacteria invasion. Lignin is also giving stiffness and rigidity to the fibres and acts as an agent to cells connection, producing an impact and compression resistant structure *(Pereira et al., 2015).*

Lignin appears in greater proportion at the end of fibre growth in the middle lamella *(Crônier et al., 2005)*. Lignin was detected in large proportion in the primary cell wall and in the outer layer of the secondary cell wall but it is rarely detected in the other layers of secondary cell wall *(Behr et al., 2019)*. Its presence can be detected by immunochemistry. Its content can be determined by the method of Klason, by weighing the residue of a treatment with sulphuric acid which dissolves the cellulose *(Tappi Standard, 2015).*

1.2.3. Development of Fibres

The formation of fibres involves stages of cell initiation, elongation, cell wall layer formation, and maturation.

The hemp bast fibres can be differentiated into two categories: primary fibres and secondary fibres. The primary fibres are originated from the pro-cambium. They first undergo a symplastic (or coordinated) growth during which the fibres extend in synchronisation with the surrounding tissues *(Gorshkova et al., 2012).* This first step occurs between 1 mm and 2 mm from the apex and fibres have flat ends *(Snegireva et al., 2015).* Then the intrusive growth takes place, the fibres spread faster than the surrounding tissues, insert themselves between neighbouring cells and the ends of the fibres are then tapered *(Ageeva et al., 2005; Hayward, 1938; Snegireva et al., 2015).* A scheme of the symplastic and intrusive growth is illustrated in Figure 1.24. The intrusive growth is the major determinant of fibre-bundle formation *(Guerriero et al., 2017).* The secondary fibres come from the vascular cambium. They raise only by intrusive growth and appear later in the development of the plant *(Snegireva et al., 2015).*

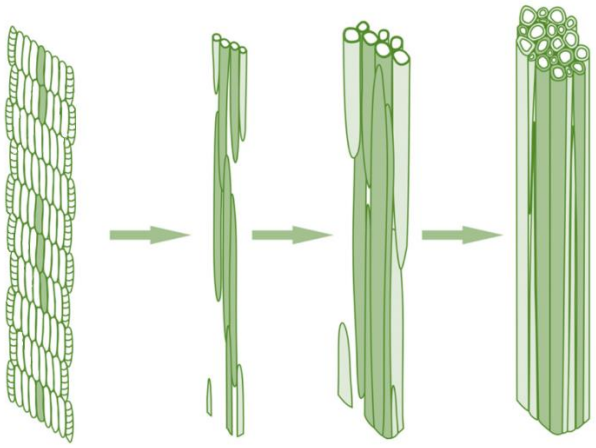


Figure 1.24: Formation of fibres from (Snegireva et al., 2015)

1.2.3.1. Fibres Maturation

Closer Look: Cell Wall Scale

Cell wall maturation proceeds in parallel with cell wall thickening and involves post-synthetic modifications *(van Dam and Gorshkova, 2003).* The thickening of the secondary cell wall begins during the vegetative phase and continues after flowering *(Snegireva et al., 2015)*. It is a longer process than cell elongation *(Snegivera 2015).* The secondary cell wall is generally defined as a spiral structure with three distinct layers: S1, S2 and S3, going from the outside to the inside of the fibre, as described previously in section 1.2.1.2 but the categorisation is a matter of debate and layers can be classified otherwise. Mellerowicz and Gorshkova (2012) and Chernova (2018) propose to define the S2 as G-layer (from "gelatinous") and S3 as Gn-layer (from "gelatinousnew") *(T. Chernova et al., 2018; Mellerowicz and Gorshkova, 2012)*. G-layer was already described for flax *(Gorshkova and Morvan, 2006a; Mikshina et al., 2013)* and for tension wood *(Gorshkova et al., 2015; Guedes et al., 2017).* Henceforth, the terms G-layer will be used in this section.

The formation of cell walls begins with the deposition of a stripped Gn-layer, and as the cell wall develops, the stripped layer is transformed gradually into a homogenous G-layer, resulting in an increase in the thickness of the G-layer *(T. Chernova et al., 2018; Kim et al., 2021).* In mature fibres, Gn-layer disappears as it is completely transformed into G-layer or it becomes very thin in the innermost region of the secondary cell wall *(T. Chernova et al., 2018; Gorshkova et al., 2004; Kim et al., 2021).* A scheme of the cell wall thickening is presented in Figure 1.25.

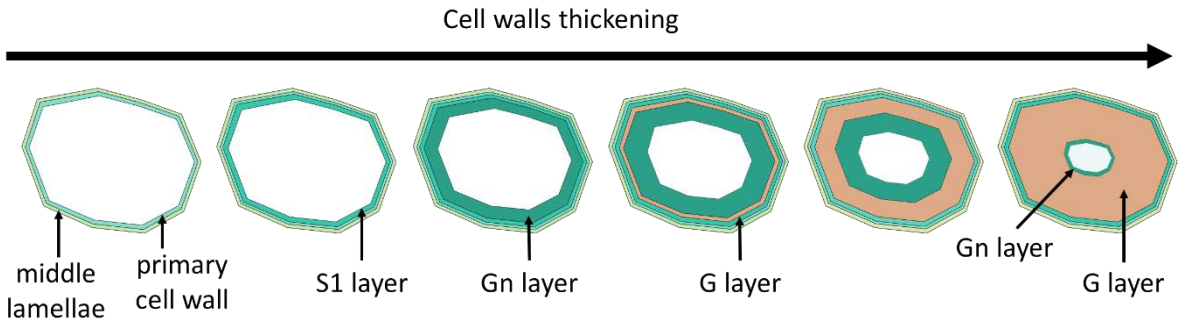


Figure 1.25: Stages of fibre thickening inspired from (Goudenhooft, 2018).

G-layer is referred to a gelatinous layer, because of its mesoporosity properties and these gel-type performances during the drying of the cell walls *(Gorshkova et al., 2012)*. It is characterised as a layer with a great content of crystalline cellulose, an orientation of the cellulose microfibrils nearly parallel to the axis of the fibre, the absence of xylan and lignin at the stage of the cell wall deposition and the presence of rhamnogalacturinan (RG-I) *(T. Chernova et al., 2018).* It has the ability to generate large contraction of the layer along the fibre axis during maturation, generating an axial strain of the fibre or axial tensile stress if the strain is prevented *(Clair et al., 2018).* The G-layer may have microfibril angles in rupture with the surrounding Gn-layer layer and may be characterised by a continuous change in microfibril angle from S1 layer to G-layer. When the change in microfibril angle is abrupt, a border line between the Gn and G-layers can be clearly observed on a cross-section *(Clair et al., 2018; Kim et al., 2021).* A detachment of the G-layer from the Gn-layer may be observed and originate from the release of the high tensile stress in the G-layer during the analysis when operating the cross-section.

The composition and transformation of fibre cell walls can be observed by light microscopy *(Gorshkova et al., 2004),* transmission electron microscopy (TEM) *(T. Chernova et al., 2018; Kim et al., 2021),* chemical reaction *(Crônier et al., 2005)*, histo- and cytochemical analysis or immunological analysis *(Blake et al., 2008; Crônier et al., 2005; Kiyoto et al., 2018)*.

With plant development, a continuous increase of cellulose content is observed, whereas at the beginning of flowering, the proportion of hemicellulose starts to decrease in contrast to that of lignin.

More precisely, the process of maturation implies a high enrichment in glucose and a variation into protein content indicating a possible role of protein in the wall network. The arabinogalactan protein (AGP) appears mainly close to the plasma membrane at the innermost surface of the fibre cell wall at flowering time *(Blake et al., 2008)* and it was observed uniformly in the S1 layer and G-layers at grain maturity *(Kiyoto et al., 2018),* suggesting that AGP deposition progresses in the whole secondary walls in the last stage of the plant development.

Immunological analysis and cytochemical staining show the presence of pectin polysaccharides in G-layers of hemp as well as in the primary cell wall and middle lamella region. Other authors suggested that maturation involves the modification of rhamnogalacturonan (RG-1), a pectin polysaccharide, by the action of fibre-specific galactosidase. In flax bast fibres, it was reported that post-synthetic modification was linked to the presence of galactans *(Gorshkova et al., 2004).* Galactans would interact with the newly formed cellulose chains of Gn-layer and the transition from the Gn-layer to the G-layer would involve a partial hydrolysis of tissue containing galactan and promotes crystallinity and microfibrils' formation *(Mellerowicz and Gorshkova, 2012; Roach et al., 2011)*. Suggested by several authors *(Blake et al., 2008; T. Chernova et al., 2018; Crônier et al., 2005; Gorshkova et al., 2010; Kiyoto et al., 2018),* a similar process as flax is happening in hemp bast fibres.

Concerning hemicellulose, it was observed that xyloglucan occurred in primary cell walls but was absent from the thick part of secondary cell walls *(Blake et al., 2008)* and is supposed to reinforce the links between the primary cell wall and secondary cell wall in the S1 layer junction *(Bourquin et al., 2002).*

Several authors reported that xylan is restricted to the primary cell walls and remain absent from the secondary cell wall *(Blake et al., 2008; T. Chernova et al., 2018; Gorshkova et al., 2015; Kiyoto et al., 2018)* while glucomannan is mainly present in the G-layers *(Kiyoto et al., 2018).* Xylan is suggested to act as a host structure for lignification, then the deposition of xylan on S1 layers may be related to the lignification of S1 layers *(Kiyoto et al., 2018).*

The lignin concentration was observed to be high in the middle lamellae, primary cell wall and the S1-layer but very low or even non-existent in the G-layers of hemp fibres *(T. Chernova et al., 2018; Kiyoto et al., 2018).* It was observed that S1-layer gets lignified especially at an advanced stage of hemp fibre development and generally the lignin content increased during fibre maturation *(T. Chernova et al., 2018; Crônier et al., 2005).* It was reported that lignin content was higher in the basal region than apical stem region; however, the analysis was including both primary and secondary bast fibres *(Crônier et al., 2005; van Dam and Gorshkova, 2003).* Since the proportion of S1 layer in total cell wall is higher in secondary fibres than in primary ones, the degree of their lignification is *higher (T. E. Chernova et al., 2018).* The lignification of the bast fibres is due to the presence of the secondary fibres rather than a modification of the primary fibres structure. In the study of Crônier et al. (2005), the lignin content of the bast fibres, including both primary and secondary fibres, remained higher in bottom part (containing secondary fibres) than top part *(Crônier et al., 2005).* Moreover, lignin characterisation highlighted that hemp fibres mostly contain guaiacyl units which are condensed lignin in nature *(Crônier et al., 2005)* and reveal an increase of less condensed lignin structure during the later stages of maturation *(Crônier et al., 2005)* which display a variation in structure compared to flax fibres *(Day et al., 2005).*

Fibres Scale

In practice, the maturity of a fibre may be assimilated and measured as a function of the size of its lumen *(Amaducci et al., 2005; Hayward, 1938)*. The smaller the lumen, the more mature the fibre. In mature fibres, the lumen, usually described as an oval shape, occupies less than one third of the cross-section of the cell *(Amaducci et al., 2005)*. It is possible to measure the maturity of fibres by a maturity index defined by Amaducci et al. in 2005, as the equation (*1-1*).

$$\mathrm{MI} = \frac{\text{mean cell wall diameter} - \text{mean lumen diameter}}{\text{mean cell wall diameter}} \mathrm{x}\ 100 \qquad (1\text{-}1)$$

Mean diameters are calculated as the average of the largest and smallest diameter measured on each cell *(Müssig and Amaducci, 2018)*. However, this method is limited by the heterogeneity of the cross-section shape of hemp fibres and the difficulty in measuring hemp fibre or lumen mean diameter as it greatly depends on the observation angle. Interestingly, the shape of the lumens in hemp is generally broad and rarely circular compared to flax which has a smaller and less apparent lumen *(Kiyoto et al., 2018).* Fibres are elongated or slightly flattened, and internal fibres are stretched further than external fibres *(Marrot et al., 2013).* From the beginning to the end of flowering, the shape of the fibres changed from oblong form to round form (*Amaducci et al., 2005)*. Another possibility for measuring the maturity of the fibres is the cell lumen percentage or porosity as stated by *(Marrot et al., 2013)* and *(Cerri et al., 2022)*, as described in the equation (*1-2*).

$$\mathrm{P} = \frac{\text{lumen area}}{\text{cell area}} \mathrm{x}\ 100 \qquad (1\text{-}2)$$

This formula can be expressed in fibre maturity calculation and be described by the ratio of the cell wall area by the entire cell wall, defined by the equation (*1-3*).

$$\mathrm{MI} = \frac{\text{Cell area} - \text{Lumen area}}{\text{Cell area}} \mathrm{x}\ 100 \qquad (1\text{-}3)$$

According to Amaducci et al. (2008), mature fibres have a maturity index greater than 65% *(Amaducci et al., 2008b)*. The maturity of the fibres is dependent on the size of the plant,

the value of the internode and indirectly to the plant density. Generally, the process of secondary cell wall deposition starts during vegetative phase of crop cycle and continues after flowering *(Amaducci et al., 2005)*. Crônier et al. (2005) observed that in basal regions the thickness of the fibres nearly reached the maximum at vegetative stages *(Crônier et al., 2005).* According to Mediavilla et al. (1998), the fibres get mature with flowering, as described in the Figure 1.26. Before flowering, the primary fibres were not very mature (80% of hollow fibres with 6 pairs of leaves and 35% of hollow fibres with nine pairs of leaves). After flowering (ten days after) 75% of the fibres are full.

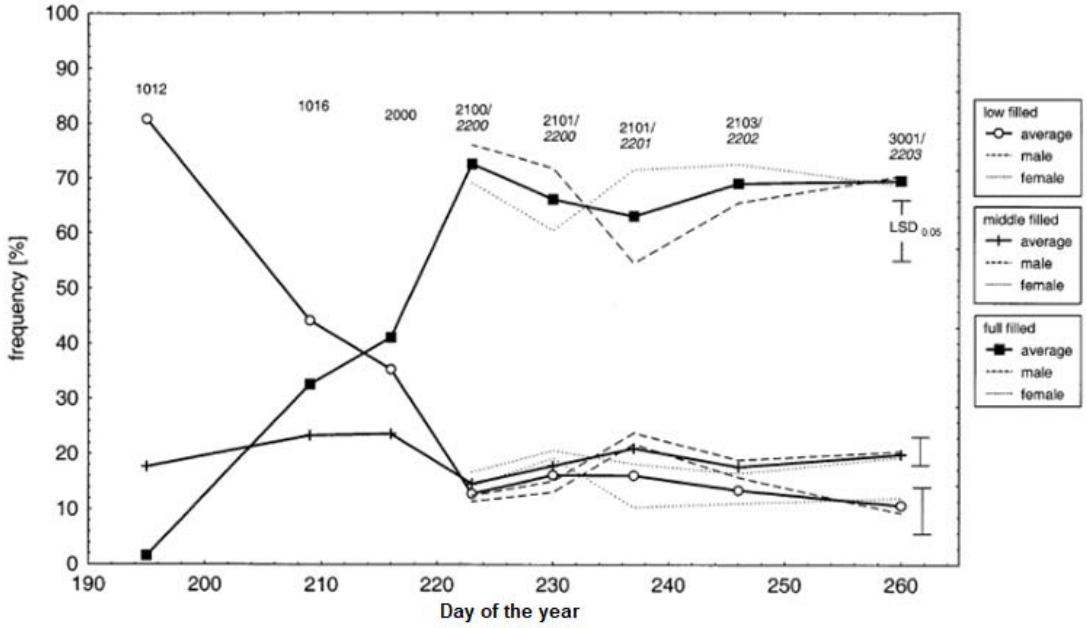


Figure 1.26: Graph from (Mediavilla et al., 1998). Filling degree of fibres distributed in the top, middle and bottom parts of the stems of male and female during growth. Decimal code of growth male/female: 2101: beginning of male flowering, 2103: end of male flowering, 2200 female flower formation, 2203: beginning of seed maturity.

According to Amaducci et al. (2005), the fibres get mature sooner in the lower part of the stem, which exhibits the oldest fibres as the fibre formation is happening at the apical part of the stem; nevertheless at full flowering the degree of maturity is similar along the stem *(Amaducci et al., 2005)*. It was also reported that the fibres become mature sooner at low density *(Amaducci et al., 2005)*. According to Müssig and Amaducci (2018), fibre maturity was statistically higher at the beginning of flowering in the highest plant density inducing larger stems diameter, whereas the situation was reversed at full flowering: the most mature fibres were found at lowest density and the least mature ones were found in the highest density *(Müssig and Amaducci, 2018).*

The diameter of the primary fibres also tends to decrease from lower to higher internodes. According to Amaducci et al. (2005), the fibres at the top are on average 15% thinner than the fibres at the bottom *(Amaducci et al., 2005)*. They reported that the diameter of the primary fibres tends to decrease from the lower to the higher plant density. Moreover, according to Amaducci et al. (2008), the extreme drought reduced stem and fibre yield, but stems had a higher percentage of fibre, finer fibres and with a higher degree of maturity *(Amaducci et al., 2008b).*

During growth, the cell walls of the primary fibres thicken from the outer fibres of the stem to the inner fibres *(Marrot et al., 2013).* According to Marrot et al. (2013), the difference in porosity between internal fibres and external fibres can differ between 6.3% and 0.65% respectively.

To summarise, the fibre filling is influenced by the harvest time, the plant density, and the genotype, from the most to least impacting parameter *(Zatta et al., 2012).*

1.2.3.2. Secondary Fibres

Secondary fibres appear during plant development later than primary fibres. They appear first from the bottom of the stems and move up to the top. They are arising from the cambium and only evolve via intrusive growth. In a stem cross-section, they are located between the xylem and the primary fibres and are arranged in the form of rings around the stem (Figure 1.27). As they develop, several layers are formed. It is possible to quantify the presence of secondary fibres by the number of layers observed and the thickness of the layer.

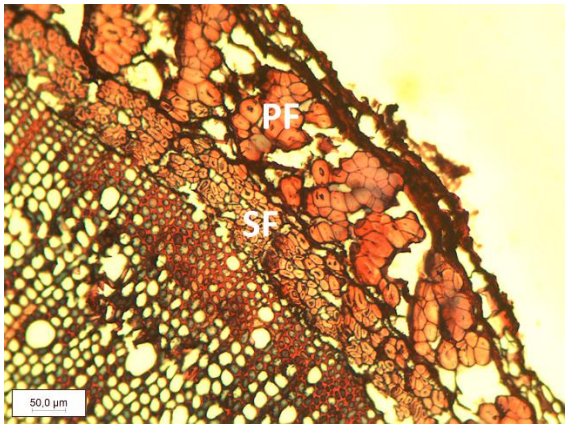


Figure 1.27: Cross-section of a hemp stem. PF: primary fibres. SF: Secondary fibres. Two layers of secondary fibres are visible.

Secondary fibres are much shorter, thinner and more lignified than primary fibres (Table 1.4).

Table 1.4: Hemp bast fibres properties.

	Primary fibres	Secondary fibres
Diameter (µm)	10–50	5–20
Length (mm)	5–50	1–2
Lignin content (%)	3.6	5.5
References	(Bourmaud et al., 2018; Fernandez-Tendero et al., 2017; Sponner et al., 2005)	(Abot, 2010; Bourmaud et al., 2017, 2017; Crônier et al., 2005; Fernandez-Tendero et al., 2017; Gorshkova et al., 2012; Linger, 2002; Placet et al., 2014b; Snegireva et al., 2015; van der Werf et al., 1994)

The appearance of secondary fibres remains unclear. Secondary fibres appear generally during the beginning of flowering time *(Mediavilla et al., 2001)* but they were observed to already grow at the vegetative stage *(Crônier et al., 2005).* Their proportion within total bast fibres fraction is increasing with the (dry) mass of the stem *(van der Werf et al., 1994)*. The bast fibres botanically belong to the sclerenchyma tissue which gives mechanical support to the plants *(McDougall et al., 1993; van Dam and Gorshkova, 2003)* and the need for such support increases when plants become heavier. When plants grow taller and the upper part

becomes heavier, due for example to the development of inflorescences or filling of the seeds, the formation of secondary fibres is more important *(van der Werf et al., 1994)* (Figure 1.28).

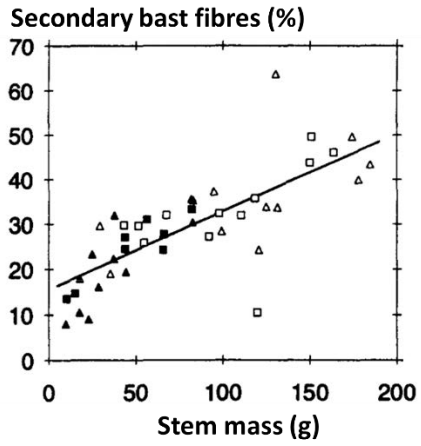


Figure 1.28: Relationship between the proportion of secondary bast fibre in the total bast fibre of the stem and the stem dry mass of the hemp plants. White symbols: plant density of 10 plants/m². Black symbols: plant density of 90 plants/m². Square or triangle depending on variety. Figure from (van der Werf et al., 1994).

According to Westerhuis et al. (2019), the height reached by the secondary fibres within the stem depends on the height and diameter of the stems *(Westerhuis et al., 2019)*. Small diameter stems have a lower secondary fibre layer thickness than larger stems (Hernandez et al., 2007a). Thus, Westerhuis et al. (2019) indicate that to obtain a maximum of primary fibres without secondary fibres, it is recommended to sow seeds at high densities and harvest around flowering *(Westerhuis et al., 2019).*

The presence of secondary fibres is dependent on the variety *(Sankari, 2000)*. Horkay (1982) found that ratios of secondary bast fibre to primary bast fibre increased with increasing bast fibre content in Hungarian high bast fibre cultivars *(van der Werf et al., 1994)*. Then breeding for high bast fibre contents tends to favour secondary fibre above primary fibre unless genotypes with high secondary bast fibre contents are eliminated *(van der Werf et al., 1994).*

Moreover, more secondary fibres were observed in a rainy year *(Sankari, 2000)* and when the xylem layer is thicker *(Hernandez et al., 2007a).*

1.2.4. Agronomic Pratices Influencing Hemp Fibres Properties

Hemp is ideally adapted to temperate climates but can, however, grow in various atmospheres *(Musio, 2018)*. In Europe, sowing is generally carried out between mid-April and mid-May (as presented in the section 1.1.3.1) *(Cosentino et al., 2012)*. Seeds are generally sown right after frost to avoid the loss of the plants and to enlarge as much as possible the growth period *(Hall et al., 2014)*. A survey in 2010, covering 99% of the production area, showed that hemp farmers in the European Union on average harvested 7.3 Mg/ha of stems *(Carus et al., 2013).* The time of harvesting is generally defined by the stage of flowering or seed maturation depending on the end use. For instance, for textile applications, hemp is largely harvested during the flowering period so that dew retting can operate with a sufficient time before the arrival of too rainy days. Agronomic techniques are highly conditioning the properties of the final hemp and the productivity. Various studies show a strong adaptability of hemp fibre's quality to different environmental conditions *(Petit et al., 2020).* The following sections detail the effects of agronomic choices and climatic conditions reported in the literature on the production of hemp fibres in terms of quantity and quality. Plant density,

nitrogen fertilisation, genotypes and harvesting time are generally the most important factors for stem yield and fibre quality.

1.2.4.1. Retting Process

For the production of long hemp fibres, the first step after harvesting is retting. Retting is a biological process that consists in eliminating the pectic substances located in the middle lamella that bind the fibres together and to the other constituents of the hemp stem (as described in section 1.1.1.3). This step is important and essential to obtain well separated fibres suitable for the spinning process *(Sponner et al., 2005).* In this section, we will focus on dew-retting. The stems are left spread out in the field after harvesting for a period which can vary from three to eight weeks. With the combined action of dew and showers of rain, the moisture content of the plants is increased. This encourages the growth of certain bacteria and/or fungi which selectively attack and remove the pectic contents in a first extent.

Dew-retting is effective if rainfall and relative humidity are high enough *(Sponner et al., 2005).* Field retting can therefore only be carried out in geographical regions where the weather conditions are suitable. The length of the retting process is critical both for fibre yield and quality *(Bennett et al., 2006).* An excessive retting weakens the fibres as the bacteria also attack the cellulosic G-layer. An under-retting does not lead to sufficient degradation of the binder between fibres. Their separation is in that case more difficult *(Jankauskienė et al., 2015; Liu et al., 2015a; Mazian et al., 2019).* As soon as microbial colonisation initiates, fungi can modify the local environment by transferring water and nutriments and facilitating the transport of bacteria participating in biofilm formation *(Bleuze et al., 2020; Ribeiro et al., 2015)*. At the early stage of retting, pectin is degraded very rapidly *(Liu et al., 2015a)*. The differences in the initial stem chemical composition due to different crop harvest time significantly affected the microbial colonisation, the biofilm formation and the retting time *(Bleuze et al., 2020)*. Bleuze et al. (2020) carried out controlled retting experiments in the laboratory and observed that the retting duration, based on the observed fibre bundle decohesion, was reduced by approximately two weeks for the hemp harvested at the flowering stage compared to that at seed maturity.

A significant change of the colour of stems and fibres is observed during retting as shown in Figure 1.29, varying between light green for unretted stem samples, yellow for low retted samples, yellow with the presence of grey dots for medium retted samples and grey for highly retted samples *(Mazian et al., 2018).* In the work of Mazian et al. (2018), the five-week field retting duration corresponding to the penultimate sample in Figure 1.29, provided the higher mechanical properties of hemp fibres, when harvested at beginning of flowering and retted in summer weather conditions in south of France *(Mazian et al., 2018).* The colour of the stems is, however,not a reliable indicator of retting quality and a global colour panel for retting evaluation would not be advised. The colour is dependent on the variety. Some varieties turn black quickly without being well retted, meaning that the fibres are not easily detached from the woody part of the stem, and other varieties are considered well retted while they are still coloured in yellow *(Blandinières et al., 2023).*

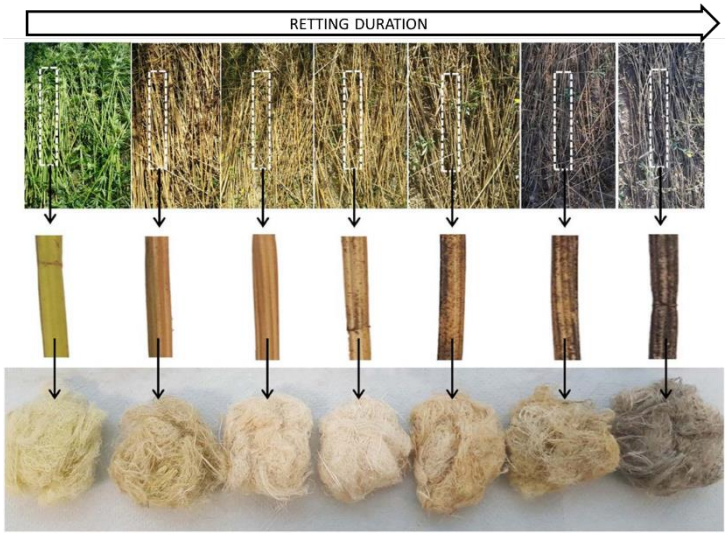


Figure 1.29: Evolution of the coloration of the stems and the fibres during retting from (Mazian et al., 2018).

Homogenous retting is important. The non-uniformity of the retting has the effect of increasing the dispersion of the properties of the fibres, for example in terms of fineness distribution and in mechanical properties of the fibres *(Liu et al., 2015a; Mazian et al., 2019).* Müssig and Martens (2003) observed that thin and wide swath produces high regularity of retting degree while a heavy swath produces a high variation in retting degree *(Müssig and Martens, 2003).* The upper part of the heavy swath was retired, and the lower part was still green. Moreover Bonnétat (1919) reported that in the past, stems were sorted by diameters: large, medium and small as they were subjected to unequal retting *(Bonnétat, 1919).* In flax cultivation, the swaths are returned once, twice or three times before the balling to homogenise the retting.

In addition to the retting process, the quality and yield of harvested hemp are strongly impacted by agronomic choices such as variety, plant density, harvest period, pedoclimatic conditions as well as fertiliser input.

1.2.4.2. Pedoclimatic Conditions

Ideally, hemp requires an average daily temperature of 16 °C to 27 °C and an average precipitation of 630 mm to 750 mm per annum *(Adesina et al., 2020).* Some studies show that hemp needs 250 mm to 350 mm of rain during the vegetative phase. Hemp roots are capable of penetrating the soil up to 2–3 m to extract moisture *(Adesina et al., 2020)*. Petit et al. (2020) suggested that a low amount of available water (196 mm of rainfall compared to 363 mm) partially causes an increase in pectin content in the stem as pectin is able to store large amount of water, and can be associated with difficulties in fibre extraction *(Petit et al., 2020).*

The most suitable soils for growing hemp have a pH between 5.8 and 7.5 are well aerated, rich in nutrients and have good water retention capacity Textures such as sandy loam soils are ideal. Seedbed conditions have a significant effect on plant establishment and influence the uniform germination of hemp seeds *(Cromack, 1998)*. Soil preparation is an essential step as any hard boots in the soil should be broken off to allow free drainage as waterlogging kill plants, especially young ones *(Adesina et al., 2020; van der Werf, 1991).*

1.2.4.3. Plant Density

The plant density of a hemp crop, meaning the number of plants per square metre at plant emergence or harvest time, is mainly controlled by the seeding rates. It has a significant

impact on the properties of the fibres in the plants *(Horne, 2012)*, the biometric characteristics of the plant and the yields *(Amaducci et al., 2002a; Tang et al., 2017; van der Werf et al., 1995)*. Seeding rate is expressed as the number seed per square metre. Seeding rates for hemp crops was found to vary widely from 50 to 500 seeds/m^2 in older recommendations *(Dempsey, 1975),* but further recommendations range from 150 to 400 seeds/m^2 *(Amaducci et al., 2008b; van der Werf et al., 1995).* The germination rate, which is a percentage comparing the density of plants at emergence and the seed density, displays large variations and is comprised between 60% and 80%. Moreover, especially at higher than 90 plants/m², plants die as a result of self-thinning. The phenomenon of self-thinning is inter-plant competition leading to the mortality of some plants *(van der Werf et al., 1995)*. As a consequence, several authors showed that increasing plant density has little impact on the biomass yields *(Amaducci et al., 2002a, 2008b; Meijer et al., 1995; Struik et al., 2000; Tang et al., 2017; van der Werf et al., 1995; Westerhuis et al., 2009).* Amaducci et al. (2002) reported that stem yield was significantly higher at higher plant density (ranging from 30 to 270 plants/m^2) in the early stage of the growth but plant density did not affect total dry matter yield at late times. In Tang et al. (2017) studies, increasing density from 30 to 120 plants/m^2 increased stem yield by 29% but further increase in plant density did not result in a significant increase in stem yield *(Tang et al., 2017).*

On another side, numerous studies examining the effects of plant density on fibre yields were carried out providing mixed results. Several authors reported that a high density enlarges the bast fibre content and hence the long fibres yield *(Heuser 1927, Jakobey 1965, from (Cromack, 1998; van der Werf et al., 1995).* By increasing plant density from 10 to 240 plants/m², the fibre content was observed to increase using mechanical extraction of the fibres from the core of the stems *(van der Werf et al., 1995).* However, other more recent studies showed that plant density has no effect on fibre content or even reduce the fibre yield while being lower *(Amaducci et al., 2008b; Höppner et al., 2004; Westerhuis et al., 2009).* In Amaducci et al. (2008) works, for two consecutive years the fibre yield at the lower density (120 plants/m²) was, higher than the fibre yield at the two higher density (240 and 360 plant/m²) using chemically extraction of the fibres *(Amaducci et al., 2008b).*

Overall, no clear relationship has been stated between plant density and stem yield, fibre content or fibre yield so far. However, changing plant density has a large effect on plant biometrics. Plant density influences the size and diameter of plants. By increasing the plant density, the plants are smaller, the stems are thinner and the basal internodes are longer *(Amaducci et al., 2002a; Hernandez et al., 2007a).* For example, it was observed in Italy that the percentage of plants above 100 cm decreased with increasing plant density *(Westerhuis et al., 2019)*. At high density, exceeding 180 plants/m², the competition for light and nutrients at the start of growth promotes internode extension *(Amaducci et al., 2002a; Musio, 2018)* but it also leads to an irregular canopy *(Zatta et al., 2012).* Moreover, at high densities the leaves tend to drop, resulting in aligned and parallel stems facilitating their harvest and processing *(Amaducci et al., 2002a; Dewey, 1913; Tang et al., 2017)*. Additionally, an optimal plant density to reach high decortication efficiency with a lab-scale breaking unit was found for 120 plants/m² *(Leoni et al., 2022)*. Khan et al. (2010) reported an increase of compression load and energy requirements as the diameter of the stem increases *(Khan et al., 2010).* Plant density has an impact on the formation and the quality of the fibres. High plant density, exceeding 180 plants/m², produces finer fibre cells *(Amaducci et al., 2002a, 2008b; Schäfer and Honermeier, 2006; van der Werf et al., 1995).* Primary fibres mature sooner at lower density and at lower internodes up to the beginning of flowering whereas at full flowering, the maturation degree was similar along the stem *(Amaducci et al., 2002a)*. Furthermore, a high density limits the development of secondary fibres *(Amaducci et al., 2008b, 2005; Schäfer and Honermeier, 2006; Westerhuis et al., 2019).* The recommended plant density, according to

Westerhuis et al. (2006), for harvesting fibres for textile purposes, regarding both the fibres quality and fibres yields, is around 180 to 270 plants/m² *(Westerhuis, 2016).*

1.2.4.4. Harvest Time

Traditionally, Asian farmers often harvest hemp fibre crops at the pre-floral stage before male and female plants can be distinguished, while European farmers usually wait until the males had shed pollen and the seeds were starting to mature before harvesting *(Clarke, 2010).* This difference in harvesting time sets the stage for the invention of two basic strategies for bark removal, fibre extraction and yarn production. The Asian strategies for bark removal and fibre extraction such as peeling, boiling and fermenting or steaming the stalks and scraping the fibres, are adapted to processing young stalks, whereas the European processing strategy of breaking the dried stalks and extracting the fibre by scutching and combing is adapted to larger and more mature stalks *(Clarke, 2010)*. In 1919, Marquart advised mowing hemp for fibres when the stem and the lower leaves of the female plants begin to yellow, and the lower leaves of the male plants are already falling, while Hoffman in 1957 mentioned that the harvest of hemp for fibres generally occurs at the end of female flowering. Spaldon and Laskos affirmed in 1964 that the highest yield of long fibres was obtained when the plants were harvested when 75% or 100% of the male plants are shedding pollen in half or more of their inflorescences *(van der Werf, 1991)*. Additionally, Jakobey stated in 1965 that if male flowering began in early July, the time of harvesting happened at its technical maturity in early August *(van der Werf, 1991).* In 2012; Zatta et al. stated that the full-female flowering stage would result in the optimal harvest time for textile use, based on studies of primary fibres softness, colour, brightness and maturity, and secondary fibre observations *(Zatta et al., 2012)*. In other studies, it was mentioned that the primary fibre content tends to decrease after flowering, mainly due to accumulation of secondary bast fibres and xylem *(Amaducci et al., 2015; Keller et al., 2001; Mediavilla et al., 2001; Salentijn et al., 2015).* Finally, the choice for harvesting hemp around the time of flowering predominates lately in Europe corresponding to the moment when the primary fibres are supposed to be homogeneously mature along the stem, lignification to be low, and fibre yield to be high *(Vandepitte et al., 2020; Westerhuis et al., 2019).* For garment textile destinations, the focus is mainly on fibre quality and fineness. Combining with a high plant density, harvesting at flowering would be advantageous for extracting fine fibres for high quality applications.

The date of harvest has an impact on the morphology of the plants and impacts the yields and quality of hemp fibres. A late harvest time induces taller and thicker plants *(Amaducci et al., 2002a; de Meijer and van der Werf, 1994; Westerhuis et al., 2019).* However, if harvest is delayed at seed maturity, yields can be reduced. From full flowering, the dry matter yield decreases *(Mediavilla et al., 2001; Struik et al., 2000; van der Werf et al., 1994).* Indeed at high plant density by delaying the harvest period, the self-thinning effect is accentuated and reduces the stem yield, as well as fibres yield *(de Meijer and Keizer, 1994; Musio et al., 2018; Tang et al., 2017; van der Werf et al., 1995)*. However, the influence of the harvest time on primary fibre content inside a plant is unclear and studies from the literature are not conclusive *(Westerhuis et al., 2019)*. Some authors reported that delaying the harvest period increases the percentage of primary fibre *(Amaducci et al., 2008b; Struik et al., 2000)* while others reported that it decreases it *(Schäfer and Honermeier, 2006)*. Additionally, the efficiency of fibre extraction is reduced by delaying the harvest. It can be a consequence of lignin deposition during the flowering period (Amaducci et al., 2002a, 2005; Musio et al., 2018).

1.2.4.5. Nitrogen Inputs

Among the nutrients consumed by the plant, nitrogen constitutes the most important contribution in the development of the plant. The effects of nitrogen are directly related to soil fertility even though nitrogen has been intensively used in fertilisers and led to large increases in crop yields *(X. Zhang et al., 2015).* Despite hemp showed limited response to N-fertilisation between 60 kg N/ha and 240 kg N/ha *(Amaducci et al., 2002b; Struik et al., 2000; Zatta et al., 2012),* the highest bast fibre content in the stem and bast fibre yield was obtained for nitrogen values around 50 kg N/ha to 150 kg N/ha *(Adesina et al., 2020; Blandinières et al., 2023; van der Werf et al., 1995).* The optimal dose resulted in 60–80 kg N/ha, which can be considered a low rate if compared to most conventional crops *(Zatta et al., 2012)*. On one hand, it was reported that nitrogen doses lower than the optimal one, caused shorter internodes and reduces the stem yield *(Struik et al., 2000).* In Tang et al. studies, increasing nitrogen fertilisation rates from 0 kg N/ha to 60 kg $N.ha^{-1}$ increased stem yield by 32% *(Tang et al., 2017).* On another hand, it has been reported that a high fertilisation reduced the cell wall thickness and cell area but increase the development of secondary fibres and led to an excessive leaf vigour, more frequent pathologies and a more irregular canopy development *(Amaducci et al., 2002b; Cerri et al., 2022),* as well as the phenomenon of self-thinning and causes plants dying and irregularities *(van der Werf and van den Berg, 1995)*. Likewise, the highest decortication efficiency was found between 0 kg N/ha and 100 kg N/ha *(Leoni et al., 2022).*

1.2.4.6. Genotypes

Monoecious and Dioecious Varieties

Various works reported differences in fibre properties between monoecious and dioecious plants, and between female and male dioecious plants, either in yields or stem and fibre morphologies.

The monoecious types have generally shorter flowering periods and higher canopy uniformity. The uniformity of the stem height is suggested to facilitate the harvest of parallel hemp stems. Nonetheless, it should be recognised that longer flowering periods are generally associated with higher yields. The results of various works suggested that the dioecious varieties tend to exhibit a higher primary fibre content than monoecious varieties *(Bennett et al., 2006; Cromack, 1998; Salentijn et al., 2019; van der Werf et al., 1994).* Zatta et al. (2012) revealed over a large panel of studies that the dioecious genotypes out yielded the monoecious ones in terms of dry biomass and fibres, and the dioecious genotypes showed a better fibre quality in terms of elementary fibre fineness than monoecious types *(Zatta et al., 2012, 2008).* The fibres yield was calculated on the base of stem yield and fibre content after chemical extraction. It is worth noting that all the dioecious varieties were intermediate or late flowering while all the monoecious varieties were early or intermediate flowering. Moreover, Angelini et al. informed in 2016 that new dioecious cultivars revealed high fibre yield after manual scutching compared to monoecious and traditional dioecious cultivars *(Angelini et al., 2016).* On the contrary, according to Sankari et al. (2000), no significant differences based on chemical analysis were found in primary bast fibres content between monoecious and dioecious varieties *(Sankari, 2000)*. Petit et al. concluded in 2020, in their works that monoecious varieties have larger fibre qualities than dioecious ones in terms of fibre bundles fineness (using a permeability airflow indicator), cell wall composition, decortication efficiency, and bast fibre content (calculated using a lab-scale roller-breaker decortication system), and explained the differences by a larger uniformity in fibre production *(Petit et al., 2020).*

In dioecious varieties, male plants reveal a significantly higher content of primary fibres compared to female plants *(Cerri et al., 2022; Rowlandson, 1849; Westerhuis, 2016).* Furthermore, it has been observed at seed maturity that male plants have more primary fibres and less thickened cell wall and smaller cells than female plants *(Cerri et al., 2022)*. However, other studies showed that male and female primary fibres filing rates did not show any distinct pattern *(Mediavilla et al., 2001).*

Concerning the presence of secondary bast fibres, the proportion was found to be higher in monoecious plants than in dioecious plants *(Amaducci et al., 2005; Crônier et al., 2005).* Female plants have a higher abundance of secondary fibres compared to male plants *(Cerri et al., 2022)* and the formation of secondary fibres begins earlier and appears with more abrupt growth at flowering for female than male plants *(Mediavilla et al., 2001).*

Periodicity

The choice of the cultivars through the critical length of their photoperiod has a significant impact on the initiation of flowering and thus on the morphology of the plants. The increase of the photoperiod leads to the formation of plants with greater height and greater diameters *(Hall et al., 2014).* When grown in optimum light duration and temperature conditions, the vegetative period is prolonged, ensuring tall stems necessary for fibre production (Hall et al., 2012).

The late-flowering varieties have a higher stem yield than the early-flowering ones *(Hall et al., 2012; van der Werf and van den Berg, 1995; Zatta et al., 2008).* Stem biomass is mainly accumulated during vegetative phase, and it was observed that late-flowering varieties grew faster during the latter part of the growing season. Then, to maximise the stem dry matter production, late-flowering varieties are rather chosen, especially in northern regions where long day lengths occur in summer and prolonged vegetative phase occurs *(Salentijn et al., 2015)*. However, by increasing the length of the vegetative phase and the size of the plants, the presence of secondary fibres might be higher. In this case, the quality of the fibres would decrease. So far, the fibre maturity showed limited variation among varieties *(Zatta et al., 2008).*

1.2.4.7. Textile

For textile applications, the dominant parameters required regarding fibre quality are fibre strength, fineness, fibre division ability, fibre length distribution and homogeneity. Fine fibres are the ones which are the most demanded for the garment textile industry. It is therefore important to grow hemp varieties cultivated and harvested with an itinerary that favours smaller fibre diameter. Thus, for textile destinations, the focus would be rather on fibre quality and homogeneity than only quantity *(Ranalli and Venturi, 2004).*

According to work published in literature, it is therefore advisable to sow at high density to obtain 180 to 270 plants/m², with a nitrogen inputs around 60 kg N/ha to 80 kg N/ha and harvest around flowering for high-quality textile end-uses. These parameters are expected to be optimum to obtain fine strong and mature long fibres without lignified secondary fibres. Moreover, in that case, self-thinning is limited, and high stem and fibre yields are expected with a good decortication ability.

1.3. Textile Fibre Preparation and Spinning

Not all natural fibres can be considered as textile fibres. A textile fibre must possess a few key mandatory properties, such as spinnability, strength, fineness or dyeability. Fibres

must be in parallel arrangement for ease of inserting a twist to form a yarn, they must have an internal attraction to give cohesion to the structure, and there should be freedom of molecular movement to provide sufficient extensibility for moisture absorption and dye uptake for coloration *(Shuvo, 2020)*. To introduce hemp fibres into the fashion textile sector, fibres should be able to form fine yarns. Most of the demand on the market targets yarns between 24 and 26 Nm. Nm is the metric number, which is the yarn length in metres per gram. The higher the Nm, the finer the yarn. The finer the yarn that can be spun, the higher the value of the material.

Two main types of technical fibres are differentiated in this thesis: long line fibres and short unaligned fibres. The long line fibres are typically extracted by scutching process and are usually spun by wet spinning to produce a fine yarn, while short fibres are generated by other extraction process or are wastes from the scutching line. They may be used to produce coarse yarns by dry-spinning or finest yarns while blended with other fibres.

1.3.1. Long Fibres Spinning

The Figure 1.30 presents the traditional wet-spinning processing route for fabric manufacturing.

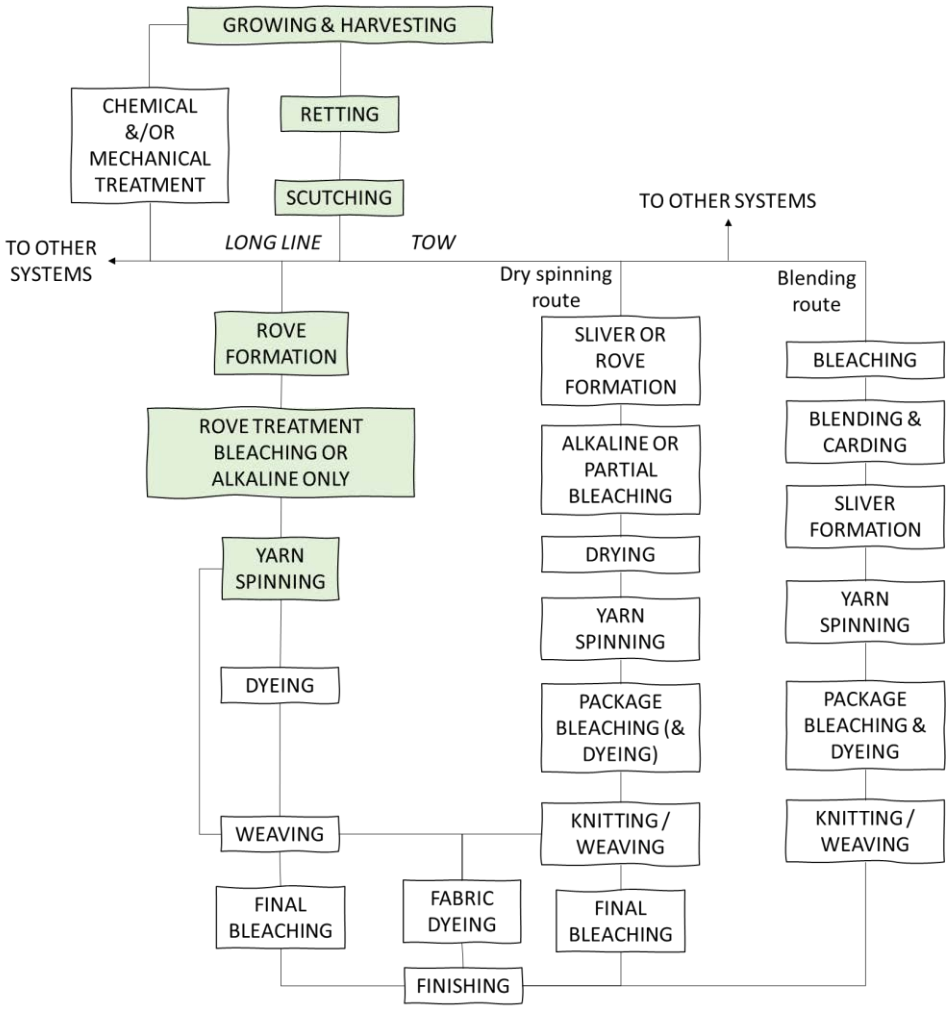


Figure 1.30: Example of processing route for fabric manufacturing inspired from (Sharma and Sumere, 1992). Traditional wet-spinning route for long fibres is highlighted.

1.3.1.1. Rove Formation: Mechanical Preparation

After the scutching extraction of the long fibres is complete, the long hemp fibres are hackled, or, in other words, combed. This process is conducted to clean the fibres from the remaining woody core and bark particles, disentangle, straighten, and reduce the technical fibre diameter. The process also removes the shorter fibres, called tows, from the long fibres. The process is conducted with the use of a hackling frame. Scutched hemp fibres are clamped in a holder and meet sets of pins of increasing fineness and density. The gradual decreasing size of the pins progressively divides the coarse fibre bundles into separated finer fibres bundles *(Klein, 2016).*

After being hackled, the long fibres are drawn and transformed into a continuous sliver. The sliver is submitted to drawing and doubling with the objective to create a uniform fine fibre sliver. Long fibre hemp slivers linear weights are typically ranging 2.5 g/m to 5 g/m. It is then processed on the roving machine to create a low twist roving that is as even as possible. The rove thread is further transformed into yarns on the spinning frame *(Horne, 2012).*

1.3.1.2. Rove Treatment: Chemical Preparation

A degumming step is generally added before the wet-spinning process to remove most of non-cellulosic impurity and help manufacturing fine yarns. In particular, it helps the removal of some pectin and lignin that are present in the middle lamellae and favours the division of the fibres. Several methods for the elimination of non-cellulosic components are existing. The chemical degumming with alkaline treatments is the most used treatment. Alkaline degumming is commonly done with sodium hydroxide (NaOH) solution. This treatment aims to remove hydrogen bonding in the network structure. This step can also be combined with bleaching.

Bleaching is a process that whiten the fibres. It involves a variety of chemical treatments and processing techniques. The non-cellulosic matters are removed from the fibres to obtain an adequate and stable white colour. Natural fibres contain colouring compounds: organic compounds, complexes incorporating metal cations, and also, a little of inorganic pigments present as surface deposits. A bleaching agent is a substance which causes lightening or loss of coloration. In particular, bleaching leads to the removal of most of the chromophores groups present in the lignin molecule. Before the 18th century, bleaching (of flax) was carried out in open space with exposure to sunlight and employed essentially crude naturally derived reagents such as alkaline potash from ashes or organics acids from fermentation of starch and milk. Bleaching consisted in laborious procedure but was not hazardous to environment *(Kozłowski et al., 2012).* In the 18th century the bleaching processes evolved with the development of chemical products and the procedure was eased. From the past century the bleaching processing is constituted by five principal operations and possible combinations:

- Alkaline boil or scour, at variable severity
- Acidic chlorination
- Sodium hypochlorite
- Hydrogen peroxide
- Sodium chlorite

The combination of the treatments varies according to the raw material and the end use. From the past century, an effective bleaching was considered when no depolymerisation of cellulose occurs, achieving high whiteness is achieved with moderate weight loss *(Sharma and Sumere, 1992)*. The chlorite treatments were reported to be the most effective, however, acidic chlorination and sodium hypochlorite are harmful methods and are also not environmentally acceptable in Europe *(Sharma and Sumere, 1992)*. Hydrogen peroxide treatments with the use of a stabilizer are the most widely used. Numerous studies were carried out to improve the

alkaline and bleaching process and reduce hazardous chemical consumption and energy consumption, by the use for example of auxiliary's products, enzymes or supercritical CO_2 *(Eren et al., 2018; Li et al., 2020; Liu and Lv, 2021; Zhang et al., 2019, 2021)*.

1.3.1.3. Wet Spinning: Ring Spun Yarns

Fine yarns from long fibres are spun with wet-spinning system. The fibres are twisted to form a continuous yarn suitable for weaving or knitting. In wet spinning system, the roving coming from the degumming treatment is passed through an ambient or hot water bath before passing through the drafting system. The scheme of the wet-spinning system is presented in Figure 1.31.

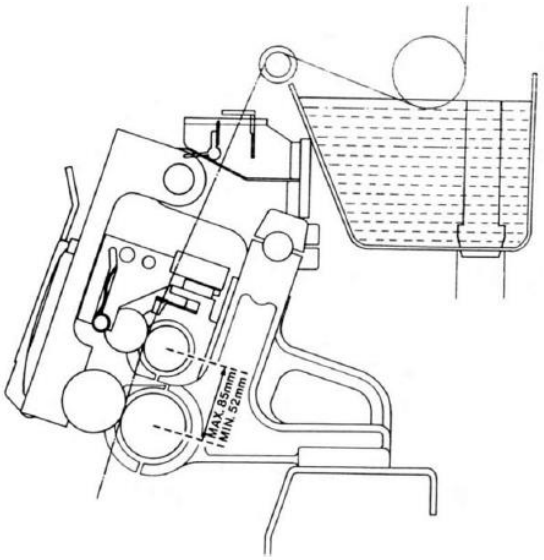


Figure 1.31: "Linmack" drafting zone for wet spinning.

In those conditions, the fibres can slide along each other's more easily. Thus, the material that binds the individual fibres into bundles are softened due to the moisture. Being wet the thread becomes smoother and more uniform than that of dry spun thread. Typical properties required for wet spinning are summarised in Table 1.5. After spinning, hemp yarn is wound on cone winders *(Renouard, 1875; Sharma and Sumere, 1992).*

Table 1.5: Properties of fibres of ring spinning from (Kozłowski et al., 2012).

Length of technical fibre (mm)	52 - 110
Fineness of fibre (tex)	0.75 - 2.2
Breaking strength (cN/tex)	19.0 - 25.0
Impurities (%)	0.2 - 1.5
Degree of polymerisation (DP)	2,000 - 3,200

1.3.2. Short Fibres Spinning

Short technical fibres in this context are fibres that are too short to be spun by wet spinning. They can be obtained from the extraction of hemp stems by means of decorticators that break the long fibres and they are also found as a by-product of long fibres extraction during scutching and hackling. These fibres may be used to produce yarns by dry spinning or yarns by rotor spinning while blended with other fibres such as cotton. The yarn preparation and spinning in a blend with other fibres with open-end rotor spinning are described in the

following section, while the dry-spinning process will not be used on this thesis and will not be detailed. For the use of open-end rotor spinning system, a cottonisation process is necessary. A cottonisation removes impurities, shortens the size of the fibres and partially or totally divides the fibre bundles. It is generally done by coupling a chemical and a mechanical treatment (Zimniewska et al., 2017). The chemical treatment is based on the same principle as the one described in section 1.3.1.2, and the details of a mechanical process are proposed below. An enzymatic method can also be used (Kozłowski et al., 2013).

1.3.2.1. Mechanical Preparation: Carding

The first mechanical processing stage for short fibre hemp prior to spinning is carding, which may be completed in a single or multiple phases. Overall, the objective of the carding process is to untangle and open lumps of fibres, remove any remaining dirt, shives and very short fibres and form a carded sliver. The carding process will parallelise the fibres and it provides an opportunity for blending different qualities of hemp or hemp with other fibres. The carded slivers are further assembled and drawn to form finer slivers before being spun. During these processes, a sizing product such as an emulsion can be added to limit frictions.

Short hemp fibres can be spun with rotor spinning system in blends with other fibres like cotton or viscose. The properties of fibres used in open-end rotor spinning, according to *(Kozłowski et al., 2012)* are described in Table 1.6.

Table 1.6: Properties of fibres for rotor spinning–open end, from (Kozłowski et al., 2012).

Length of technical fibre (mm)	24 - 32
Fineness of fibre (tex)	0.4 - 0.8
Breaking strength (cN/tex)	14.0 - 18.0
Impurities (%)	0.0 - 0.1
Degree of polymerisation (DP)	2,000 - 3,000

1.3.2.2. Open-end Rotor Spinning

Rotor spinning process was firstly commercialised in the ’60s and has continuously developed. It was aimed at providing a cheaper spinning system than the conventional ring spinning system through its higher productivity. Rotor spinning eliminates the need for a roving, since rotor yarns can be spun directly from drawn slivers. Production rate in 2010 could reach 200 m/min while in the ring spinning it was about 40 m/min *(Das and Alagirusamy, 2010).*

It is a high-speed spinning process, producing large yarn volume, with wide adaptability, and can be practically fully automated, however, the quality of the produced yarns is still not matching that of conventional ring spinning. The main emphasis of rotor-spun yarns is in the range of coarse and medium yarn count, between Nm 10 (100 tex) and Nm 62 (16 tex) while ring spinning excels in the medium to fine yarns count (finer than Nm 62 (16 tex)) *(Das and Alagirusamy, 2010).* Applications of rotor spun yarns include denim, towels, blankets socks, t-shirts, shirts and pants.

Open-end rotor spinning is a technology for creating yarn without using a spindle, on the opposite to ring spinning. Once the sliver is formed, it is fed into a spinning rotor, which consists in a hollow cylindrical structure. As the rotor spins rapidly, centrifugal force drags the fibres away towards the outer edge of the rotor, as illustrated in Figure 1.32. The rotor contains a small opening though which the fibres emerge. They are stretched and twisted to form a continuous yarn. The yarn is then wound onto a bobbin above the rotor.

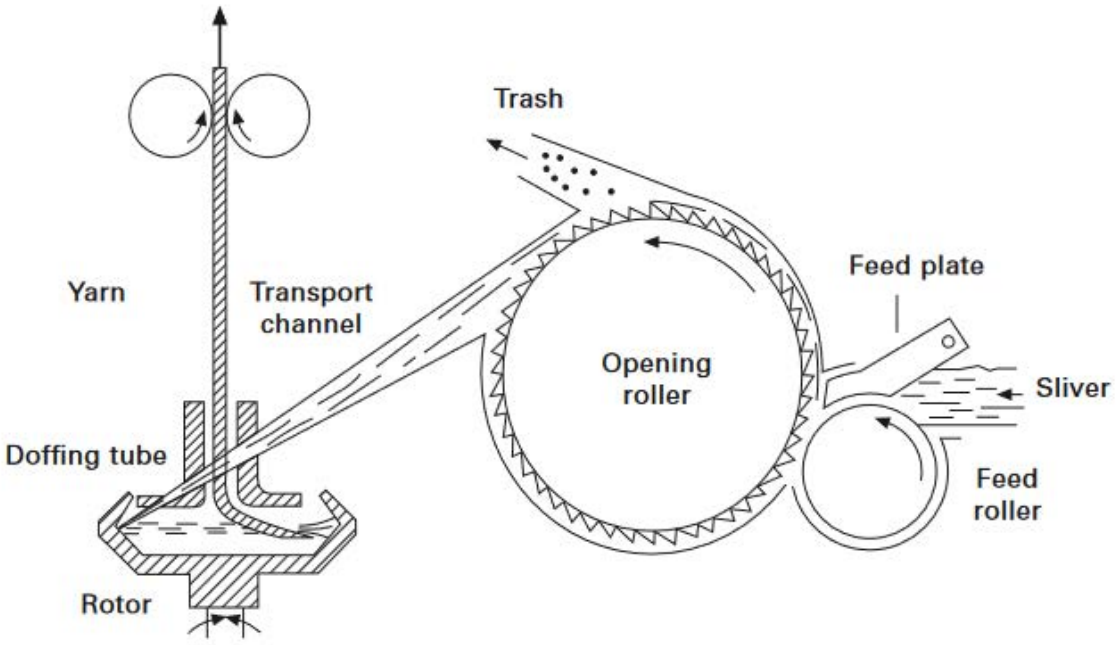


Figure 1.32: Main features of the rotor spinning system, from (Das and Alagirusamy, 2010).

For example, in Kozlowski et al. works, short hemp fibres removed during the hackling process were cottonised with enzymes before blending with 30%, 40%, and 50% of cotton. Cotton–hemp-blended yarns were produced by rotor spinning with linear mass of 32, 40, and 50 tex for the 70/30 blend, 50 tex for the 60/40 blend, and 100 tex for the 50/50 blend *(Kozłowski et al., 2013).*

1.3.3. Yarns Characteristics

The characteristics of a yarn are strongly dependent upon the characteristics of its fibres, but also upon the structure of the yarn itself. The following factors are especially significant: the number of fibres in the yarn cross-section; the fibre disposition and alignment, the position of the fibres in the strand (in the core and in the sheath), the twist. The more flexible a fibre, the more twist it can undergo and the stronger a yarn will be produced. If the fibres are stiff, they will break down while twisting *(Shuvo, 2020).* The number of fibres in the yarn cross-section determines, among other things, strength, evenness, insulating capacity and the spinning limit of the raw material. Additional fibres in the cross-section provide not only additional strength but also better evenness in the yarn. Finer fibres have more cohesion and so require less twist to form a strong yarn *(Saville, 1999)*. About thirty fibres are needed at the minimum in the yarn cross-section, but there are usually over 100. One hundred is approximately the lower limit for almost all new spinning processes *(Klein, 2016)*. The fibre length has an influence on fibre strength as well as on the spinning limit. The longer fibres, within a length limit, require less twist to cohere together to impart strength to yarn *(Morton et al., 2008).*

During ring spinning, the twisting takes place from the outside inwards. Fibres take up a helical orientation in the core and in the sheath. They can possess noticeable hairiness on the yarn surface mainly caused by sticking shorter fibres *(Klein, 2016).*

In contrast to ring spinning, twisting during rotor spinning takes place from the inside outwards. The yarn consists of fibres wrapped around the surface of the yarn *(Ernst 2016)*. The fibres from the interior are more compact while the fibres from the exterior are less twisted and place

more randomly. The fibres have a lower degree of parallelisation and the lower degree of straightening causing a lower strength of the rotor-spun yarn relative to ring-spun yarn *(Klein, 2016).* The yarn from rotor spinning may have a lower quality compared to ring spinning methods, but it offers advantages in terms of cost effectiveness and productivity.

A comparison of a ring-spinning yarn and an open-end rotor spinning yarn are presented in Figure 1.33.

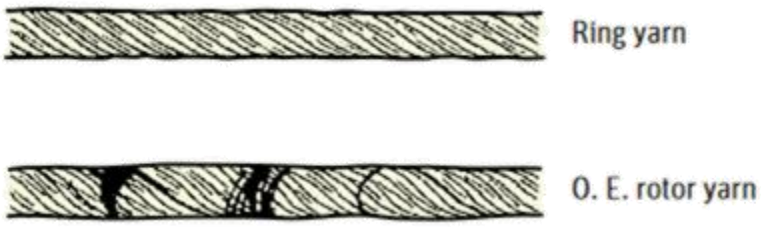


Figure 1.33: Yarn structure a ring spinning and an open end (O.E.) rotor spinning processes (Klein, 2016).

1.4. Conclusions

Hemp is an ancestral plant that was cultivated for millennia by humankind particularly for the fibres in its stem. It is arousing renewed interest this past decade mainly for its low environmental impact. The hemp plant is harvested for its flowers, its seeds and its stem and is used for many applications in construction, animal and human feed, paper industry or pharmaceutical and medicinal industries. For garment textile purpose, fine, resistant, and homogenous fibres are desired as well as a high fibre yield.

The growth stages of the fibres are quite well described in literature and are very similar to flax fibres. The process of cell initiation, cell wall thickening, and post-synthetic modification are detailed in many studies. Contrary to flax plants, hemp has the particularity to development a secondary system. It develops secondary fibres that are not desirable by spinning companies to produce high-quality yarns. The presence of secondary fibres is in relation to the morphology of the stems. It is desirable to sow with high density and control nitrogen inputs to obtain thin stems, low content of secondary fibres and a high content of primary bast fibres. The agronomy of hemp for fibre cultivation was the subject of numerous studies. In north of France, a sowing date around April and beginning of May is rather advised with a harvesting date at the beginning of August. Once the stems are cut, they are laid on the field for dew-retting. Some areas are more prone to retting than others because the process is highly dependent on climatic conditions and requires an alternance of rainy and sunny weather. One major factor impacting fibre quality is the choice of the variety. Hemp is photoperiodic and varieties can be described as early or late flowering. The late-flowering varieties are known to produce a higher stem yield as they have a longer vegetative phase. Many studies are comparing the fibres varieties from gender: dioecious or monoecious varieties, but results remain unclear. The dioecious varieties are the natural state of hemp, they were used in the past and are considered promising for textile applications, while monoecious varieties would be better suited to dual-purpose goals. Nevertheless, it remains difficult to find dedicated varieties for an adapted area and application. A lack of response is still occurring regarding the development and the quality of fibres depending on the types of varieties. Seed breeders are improving their selection process and developing new varieties for specific characteristics.

Moreover, it remains a lack of knowledge regarding the hemp processing with recent mechanisation. Large advancements have been done those recent years on the development of new harvesting machines. A system that cut the stems in two portions at a desired height and laid down the stems aligned and parallel in the field was sought. This process favours the dew-retting of the plants and produce stems that are adapted to further extraction process based on flax scutching lines. Preliminary studies demonstrated the extraction possibility of hemp stems with flax scutching; however, parameters need to be adapted. The flax long scutched fibres are further process by wet spinning. The wet-spinning process is adapted to long fibres and is generally used to produce fine yarns for garment textiles. Another spinning process is adapted for shorter length fibres and by blending two types of fibres, it forms yarns with combined properties.

An approach with large-scale trials would support the development of industrial textile hemp in France and Europe and participate to local reindustrialisation at the same time as providing potential solutions current climate changes.

Chapter 2.
Hemp Varietal Selection

This chapter focuses on the varietal selection of hemp for high-value textile applications. Industrial hemp can be divided into several groups according to their biological characteristics. According to the sexual system, hemp can be monoecious or dioecious and according to the photoperiodicity, hemp can be categorised as early or late flowering. This chapter aims at providing knowledge of hemp varieties to optimize the selection of variety for the production of textile fibres.

First of all, the dioecious character of hemp is investigated through the study of fibre properties from male and female plants. The fibre fineness, the mechanical properties and the long fibre yield are examined.

Secondly, an extensive study of the interest of five hemp varieties for textile fibre production in Normandy is described. The photoperiodicity and the gender are considered, and the optimal harvesting time for obtaining fibres suitable for fine yarn manufacturing is investigated. The cultivation of several varieties was monitored during three successive years. The development of the bast fibres during the plant development was characterised by numerous histological cross-section observations and finally the properties of the extracted fibres are detailed. Characterisations in terms of fibre yields, fibre bundle fineness, mechanical properties and organoleptic properties are studied.

2.1. Introduction

Hemp is a multifunctional crop with low environmental impact; it is grown for its seeds, its flowers, its leaves and finally for its fibres. The area under cultivation in Europe is in continuous increase *(Interchanvre, 2020; Turunen, 2006; Visković et al., 2023b).* The most valuable product in fibre dedicated hemp is long aligned fibre bundles spinnable by wet spinning process and used for garment textiles *(Ranalli and Venturi, 2004; Sponner et al., 2005).* To obtain those long-aligned fibre bundles, agricultural equipment dedicated to hemp are in development in Europe aiming at a complete, mechanical, and optimal process for harvesting, dew-retting, scutching, hackling and wet-spinning, based on the traditional linen process *(Müssig et al. 2020; Vandepitte et al. 2020; Zimniewska 2022; Pinsard et al. 2023).* The quality of the obtained spun yarn is directly impacted by the state of the raw fibres. The dominant parameters required in textile applications are fibre divisibility (separation potential of the fibres within bundles), strength and fineness (thickness of fibre bundles) *(Ranalli et Venturi 2004).*

To reach the objectives of textile quality requirements, the stems need to be well retted to favour a good divisibility of fibres, and fibres need to be sufficiently matured to possess good tensile properties *(Richely et al., 2021).* The concept of maturity here corresponds to thick secondary cell walls associated to a very low size lumen and post-synthetic modification *(T. Chernova et al., 2018; van Dam and Gorshkova, 2003).* Measuring the proportion of the cell wall area inside the fibre cell is considered as a good indication of the growth of the fibres *(Amaducci et al.2002; Marrot et al. 2013).*

The valuable fibres are bast fibres, part of sclerenchyma tissue on the outer side of the stem and are called primary fibres *(Aloni 1987)*. They are derived from the pro-cambium, measure around 5 mm to 50 mm long *(Sponner et al., 2005),* are mainly composed of cellulose and possess a low amount of lignin. A second bast fibre present in hemp is called secondary bast fibre. The latter are originated from the cambium and are more lignified than the primary fibres. They are about 2 mm long and appear later in the development of the plant *(van der Werf 1994; Crônier, Monties and Chabbert 2005; T. E. Chernova et al. 2018)*. Those shorter fibres, grouped in bundles, are expected to be separated from the long fibres during the scutching process and constitute a large part of the scutched tows that can be valorised in

technical applications. However, if they remain tied to the long fibres after extraction, they would decrease the quality of the extracted fibres leading to less homogeneity, and would hamper high value yarn spinning *(Westerhuis 2016)*. They are described as too short for spinning and have a high lignin content that could prevent the division of fibres and break the sliver during spinning, or can engender itching fabrics when combined into the yarn. They have been found in technical fibres after extraction with roller mill machine *(Fernandez-Tendero et al., 2017)*. As a consequence, it is often said that it would be beneficial when dealing with textile purposes to grow hemp plant which only possesses primary fibres.

The development of the bast fibres is directly affected by agronomic factors *(Amaducci et al. 2002).* The maturity of the fibres is reported to be dependent on the size of the plant, the value of the internode and indirectly to the plant density, nitrogen inputs or climatic conditions *(Mediavilla et al. 2001; Amaducci et al. 2008; Marrot et al. 2013; Leoni et al. 2022)*. Secondary fibres appear when the plants need strength to support their weight. So this appearance depends on the diameter and the height of the plant *(Westerhuis et al., 2019).* The proportion of secondary bast fibre in the bast fibre fraction increases with dry stem weight *(van der Werf 1994).* A high sowing density would avoid secondary fibre formation *(Amaducci et al.2005)* as it promotes lower diameter and therefore lower stems mass. Furthermore, it has been reported that filling of fibres and presence of secondary fibres occurs around flowering time *(van der Werf 1994; Mediavilla, Leupin and Keller 2001).* Flowering time fluctuates with variety and latitude as hemp is a photoperiodic plant. The earliness of the flowering is expected to have a direct effect on fibre development for a given location.

Evaluating the harvest time to obtain quality hemp fibres for textile application is biologically a balance between limiting the development of secondary fibres and optimising the filling rate of primary fibres. After being harvested, stems need time for dew retting. The retting process is highly controlling the divisibility of the fibres bundles and separation of the fibres bundles from the woody part. Dew retting is dependent on weather conditions and occurs in periods with an alternance of precipitation and dry weather. After retting, the field should not be too muddy for the tractor to be able to work in good conditions and for the stems to be collected in a dry state for preservation. These requirements impact the flexibility in harvest timing as the level of humidity rises after the beginning/mid of September in area such as Normandy in France. The risk of baling humid stems increases after this time, and it raises the risk of losing stems from rotting.

The appearance of secondary fibres and the filling-up of secondary cell walls in primary fibres may be different for each variety, and it is therefore crucial to determine the best compromise between primary fibre filling and secondary fibre appearance. Flowering is considered as a key moment at which the primary fibres are rather well filled without much appearance of secondary fibres. However, no real screening was performed to assess if some varieties lead to a better balance than others. As an example, it is usually considered that growing late-flowering varieties favour high biomass yield but the filling up of primary fibres and the quantities of secondary fibres were not determined *(Salentijn et al. 2015; Hall et al. 2014).*

Improvement in genetics from the last decades allows the development of new varieties of hemp with different photoperiodicity in order to improve the bast fibre content *(Salentijn et al., 2015)*. For further information regarding the breeding control and genetic component of hemp for fibre quality, flowering control or sex determination, the reader can refer to Salentijn et al.'s work *(Salentijn et al., 2019, 2015)*. For breeders and farmers, a variety is a plant selected with fixed agronomic characteristics or commercial interest. The plants can be reproduced by seed multiplication to obtain the same characteristics. A variety can be registered in the official seed catalogue. A cultivar is a plant that comes from human selection

work and is still in the selection process. In this chapter and the next ones, the term variety will be rather preferred in order to avoid any confusion and reflect a plant selected by breeders that is already on the official seed catalogue or still being selected. For textile applications, varieties with a high long fibre yield, a high fibre bundles fineness, fibres with strong tensile properties and fibre homogeneity are the main targeted traits *(Bonatti et al., 2004; Interchanvre, 2017; Kirk et al., 2023; Petit et al., 2020).* Those characteristics may vary with the photoperiodicity and gender.

The objective of the work is to evaluate the influence of harvest time, gender and flowering period of plants on fibre quality. In a first part, the dioecious character of hemp is investigated through the study of textile fibre characteristics from male and female plants. The fibre fineness, the mechanical properties and the long fibre content are examined. The existing instruments and standards for evaluating the fibre quality differ among studies in the literature and are not fully adapted to hemp fibres yet. The materials and the methods used in this chapter will be presented in this first study. Secondly, an extensive study of several varieties in Normandy is described. The cultivation of varieties having different photoperiodicity was monitored during three successive years. The development of the bast fibres during the plant development was characterised by numerous transverse view observations and finally the properties of the extracted fibres are detailed. Characterisations in terms of fibre yield, fibre bundle fineness, mechanical properties and organoleptic properties are studied.

2.2. Studies on Dioecious Varieties

The hemp plant is naturally dioecious, meaning that male and female flowers are borne on separate plants. It was considered that bast fibres from dioecious varieties would be more suitable for textile applications than monoecious varieties, especially because male plants are believed to contain finer fibres and a higher percentage of primary fibres. Monoecious hemp has male and female flowers on a unique plant. This type of hemp can occur naturally in exceptional stress conditions. Breeders used those atypical hemp to develop new varieties and create crops with more homogeneity that were required in the context of industrial development and mechanisation of harvest. In the past, dioecious hemp were used; male plants were reported to have finer fibres and were used for the finer household purpose, whereas fibres from female plants were used for coarser fabrics *(Bonnétat, 1919)*. It should, however, be noted that the male plants were generally harvested as soon as their pollen shed and the female plants were harvested when the seed was riped *(Rowlandson, 1849).*

According to recent literature, at seed maturity the male plants revealed a higher content of primary fibres and finer fibres compared to female plants and they also develop less secondary fibres *(Cerri et al., 2022).* It was reported that female and male plants a exhibit similar pattern of cell wall thickening, but also that the female plants display a more thickened cell wall at seed maturity *(Amaducci et al., 2008b; Cerri et al., 2022; Mediavilla et al., 2001).* Differences in cell maturity and fibre content were also reported to vary according to the height of the sample within the stem. During plant development, fibre maturity was reported to decrease from the bottom to the top of the stem and the diameter of the primary fibres also tends to decrease from the lowest to the highest internodes *(Amaducci et al., 2005).* It was also reported that the thickening of the cell wall influences the mechanical properties. The smaller the lumen, the higher the tensile properties *(Richely et al., 2021)*. Hence, the mechanical properties might also differ with gender and sample height.

The objective of this study is to provide more information on the properties of the extracted fibres in relation to sex and location within the stem. Three different dioecious varieties were studied. The three varieties were cultivated in three different sites and harvested at different times:

- Djumbo-20 is an early-flowering variety. It was harvested at full female flowering for two consecutive years. The male plants were already dried; and this does not favour dew retting.

- Carmagnola is a late-flowering variety. It was cultivated and harvested in 2020 in South-west of France. Female plants were dew retted and male plants were stand-retting, meaning that they were left standing on the field after senescence. They were pulled up separately. The quality of retting according to organoleptic tests and previous observations were satisfactory. Plants were approximately 160 cm high. The roots were removed, and the stems were manually cut in two portions of 80 cm. Stems portions are presented in Figure 2.1.

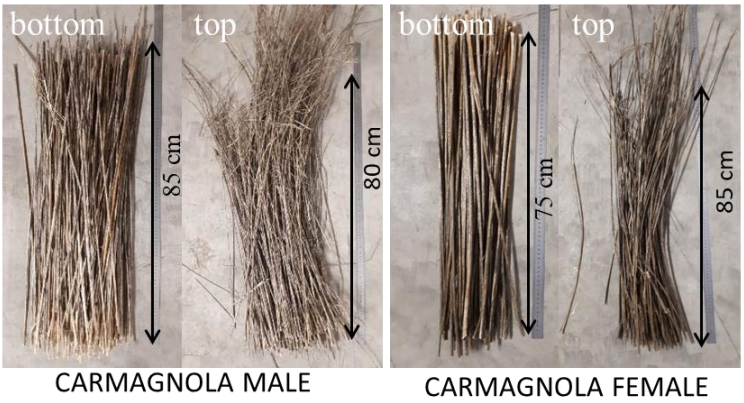


Figure 2.1: Carmagnola plants after retting, cut in two portions.

- Tiborszallasi is a late-flowering variety, harvested at female full flowering and dew-retted. Male and female were harvested at the same time and mixed together. Top and bottom parts were separated.

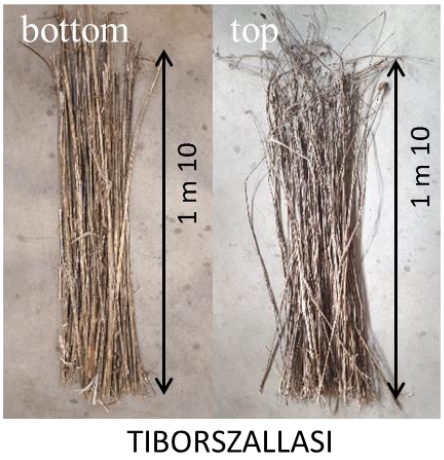


Figure 2.2: Tiborszallasi plants after retting and cut in two portions.

Three important fibre properties for textile applications were compared: the long fibres content, the fibre bundles fineness, and the elementary fibre mechanical properties. Additional studies on stem cross-sections were investigated.

2.2.1. Long Fibre Content

The three batches were extracted with a scutching and hackling laboratory device. The stems were attached to a clamp and were processed on one side and then reversed. The Figure 2.3 presents a sample processed on one side.



Figure 2.3: Half part of hemp stems processed by the scutching and hackling laboratory machine Taproot.

Laboratory Extraction Process:

A scutching-hackling device developed by Taproot Fiber Lab (Nova Scotia, Canada) is used. It is composed of three distinct modules as shown in Figure 2.4 and Figure 2.5. The first step involves breaking the wooden part of the stem into small pieces. For this, the stems are introduced vertically into the device and driven between three successive counter-rotating pairs of fluted rollers arranged in staggered rows. The pressures applied by the cylinders on the material are adjustable by means of springs constrained by pressure screws. The level of this pressure depends on the level of crushing desired, the type of material as well as the diameters of the tested stems. It is also possible to adjust the rotation speeds of the rollers. A large part of the shives and plant dust is extracted during this operation.

The material is then transferred to a second module where it is beaten. Two blades rotating in opposite directions strike the crushed stems alternately to bring down the pieces of shives and plant dust still entrapped in the fibres. In this module, the material is struck and shaken, which favours a better extraction of the waste. Beating duration and speed are adjustable.

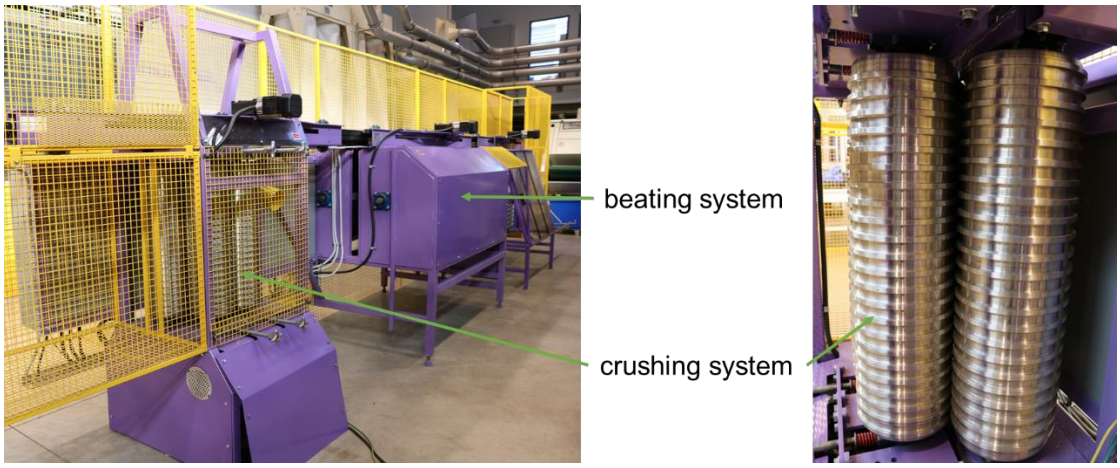


Figure 2.4: Lab-scale scutching device from Taproot Fiber Lab (Nova Scotia, Canada) including a crushing device and a beating device.

Finally, the fibres are subjected to progressive hackling. Combs, with decreasing spacing between the pins, are mounted on rotating tracks. Wide combs are necessary to align and untangle the fibres, and the progressive refinement of pins increase the fibre bundle separation and therefore a material refinement. Combing time and speed are adjustable. In the Taproot device, the combs of the two opposing caterpillars are interpenetrating so that the material is combed through its entire thickness. For the extraction of hemp, smooth parameters are used.



Figure 2.5: Combing system from Taproot Fiber Lab (Nova Scotia, Canada).

Results:

The Table 2.1 presents the long fibres contents of the Carmagnola variety after scutching and hackling.

Table 2.1: Carmagnola long fibres content after lab-scutching and hackling, according to gender and height.

Carmagnola Batches	Long hackled fibres content (%)
Female Bottom	10.8 ± 0.8
Female Top	6.2 ± 0.9
Male Bottom	12.3 ± 0.8
Male Top	6.5 ± 1.1

In this first batch, it is observed that the bottom parts have higher fibres content than the top parts and the content of the male batch is higher than the female one.

The Table 2.2 presents the long fibres contents of the Djumbo-20 variety after scutching and hackling.

Table 2.2: Djumbo-20 long fibres content after lab-scutching and hackling, according to gender and height. Results from hemp cultivated in 2021 and in 2022.

Djumbo-20 Batches	Long hackled fibres content (%) 2021	Long hackled fibres content (%) 2022
Female Bottom	9.5 ± 1.1	12.4 ± 1.3
Female Top	10.5 ± 1.3	
Male Bottom	9.3 ± 0.9	15.6 ± 1.5
Male Top	9.2 ± 1.4	

In this second batch, each sample revealed very close content for hemp cultivated in 2021 whereas a higher fibre content is observed for the male batch in 2022.

The Table 2.3 presents the long fibre contents of the Carmagnola variety after scutching and hackling.

*Table 2.3: Tiborszallasi long fibres content after lab-scutching and hackling, according to the height. *contains much residue.*

Tiborszallasi Batches	Long hackled fibres content (%)
Bottom	14.5 ± 0.9
Top	17.3 ± 1.5 *

In the third batch, the top part revealed a higher content than the bottom part but it contains larger residues. The distribution of the stem diameter was centred 9.1 mm for the bottom part and 5.7 mm for the top part and might have an influence on the decortication efficiency.

2.2.2. Mechanical Properties of Elementary Fibres

The mechanical properties of hemp elementary fibres were carried out by tensile tests. At least 30 samples of each variety were tested.

Tensile Test:

Elementary fibres were extracted manually by dividing the fibre bundles under a magnifying glass from hackled batches. Each single fibre (of a diameter lower than 20 µm to minimize the possibility of testing a very small fibre bundle) was positioned between two plastic tabs with a 12 mm gauge length and glued with a UV-curing adhesive (DYMAX ultra-light weld, Wiesbaden, Germany). The cross-section areas of each elementary fibre were determined using an automated laser scanning device, the fibre dimensional analysis system (FDAS), coupled with UV Win software (Dia-Stron Ltd., Hampshire, UK). The fibres were measured with the use of a high-precision laser photodetector (LSM-500S, Mitutoyo, Japan). Diameters were determined with the accuracy of 0.01 µm. The fibre was rotated and scanned at 10 different positions along the fibre (

Figure 2.6).

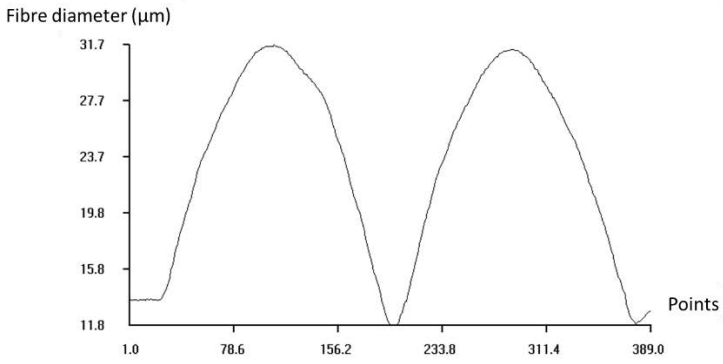


Figure 2.6: Dimensional analysis of elementary fibres by FDAS.

The maximum and minimum diameters were recorded for each section, during a full rotation of the sample along its axis. The cross-section area of the fibre was then calculated

following an elliptical model. With such model, an area close to the real area of the single fibres, which are predominately not cylindrical, may be evaluated with a minimum of error. A mean value of the cross-section area along the fibre was then computed.

Tensile testing was performed according to an improved method from the standard ASTM D3822/D3822M–14, standard test method for tensile properties of single textile fibres (ASTM D3822/D3822M–14, 2020) using the computed elliptic cross-section area. Elementary fibres were mechanically tested under tensile testing with an automated Lex high-precision extensometer (LEX820, Dia-Stron Ltd., Hampshire, UK). The fibres were loaded at a crosshead displacement speed of 1 mm/min. The displacement was obtained with an accuracy of 1 µm. The measuring points were recorded with a periodicity of 20 ms. A curve of force as a function of the displacement was obtained, and the tensile strength, the modulus of elasticity, and the failure strain could be calculated. Two strain intervals were used for stiffness calculation, as illustrated in Figure 2.7.

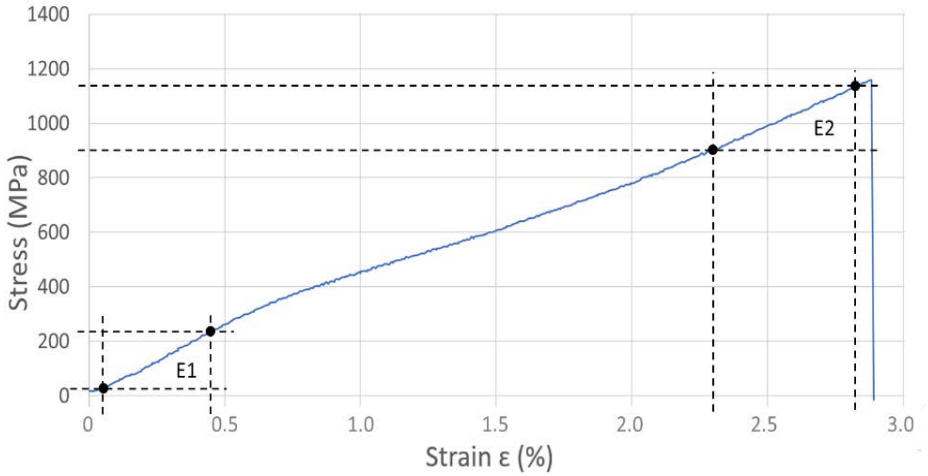


Figure 2.7: A tensile stress–strain curve of an elementary hemp fiber, with the representation of two intervals used for the calculation of tensile moduli E1 and E2.

Results:

Table 2.4 presents the tensile properties measured with the method described above. The tensile modulus E1 were calculated.

Table 2.4: Tensile properties of elementary primary fibres from the three varieties. Statistical Student test is described in Annex 3: letter 'a', 'b' and 'ab' are used to report the results of the pairwise comparisons within the same variety.

Tensile properties of Carmagnola variety.

CARMAGNOLA Variety	MALE bottom		MALE top		FEMALE bottom		FEMALE top	
Strength at break (MPa)	624 ± 261	*a*	624 ± 250	*a*	727 ± 343	*a*	687 ± 345	*a*
Tensile Modulus (GPa)	29.7 ± 11.0	*a*	32.3 ± 11.7	*ab*	37.7 ± 14.1	*b*	32.6 ± 11.9	*ab*
Strain at break (%)	2.3 ± 1.0	*a*	2.0 ± 0.8	*a*	2.0 ± 0.7	*a*	2.2 ± 0.6	*a*

Tensile properties of Djumbo-20 variety.

DJUMBO-20 Variety 2021	MALE bottom		FEMALE bottom		MIX bottom (late harvest)	
Strength at break (MPa)	742 ± 415	*a*	765 ± 271	*a*	809 ± 405	*a*

Tensile Modulus (GPa)	39.1 ± 17.0	*a*	42.5 ± 13.0	*a*	40.5 ± 13.7	*a*
Strain at break (%)	2.5 ± 0.7	*a*	2.3 ± 1.1	*a*	2.4 ± 1.0	*a*

Tensile properties of Tiborszallasi variety.

TIBORSZALLASI Variety	**bottom**		**top**	
Strength at break (MPa)	749 ± 304	*a*	465 ± 222	*b*
Tensile Modulus (GPa)	47.8 ± 16.6	*a*	37.8 ± 15.9	*b*
Strain at break (%)	1.6 ± 0.4	*a*	1.3 ± 0.4	*b*

In the first two batches, fibres from female plants show higher mechanical properties than the ones from the male plants in terms of strength at break and tensile modulus. A significant difference in tensile modulus is observed between fibres of male and female bottom part of Carmagnola variety. Moreover, it is observed that by delaying harvest of the Djumbo batch, higher fibres strengths at break were obtained. It seems that the fibres are still maturing and continue to thicken their cell wall after female full flowering.

Finally, in Tiborszallasi batches, (collected at full-flowering) the bottom part has higher tensile properties than the top part. Those result can be correlated with previous work in literature reporting that the fibre maturity decreases from the bottom to the top of the stem *(Amaducci et al., 2005)*.

2.2.3. Fineness

Fibres fineness is an important property for the textile industry in order to qualify the material and form fine yarns. It corresponds to the distribution of the fibre bundles diameter. The term fibre diameter improperly refers to fibres with circular cross-sections, which is obviously not the case with natural fibres in general and hemp fibres in particular. Later, the terms thickness and width are indistinctly preferred in order to avoid any confusion and reflect the average width along the fibre bundle's length in a batch. Usually, in textile industry, thin fibres are called fine fibres and thick fibres are called coarse fibres.

Fineness Measurement:

Some methods and instruments are described in the literature to measure the thickness of the fibres, such as Airflow test method, Sirolan-Laserscan, FMT-Shirley device or Optical fibre diameter analysis (OFDA) *(Baxter, 1997).* They are originally used for wool analysis *(Grishanov et al., 2006)*. Laserscan is a system based on a laser scan technology and requires the material to be cut in 2 mm portions. The material is dispersed in an alcoholic liquid and carried by the fluid flow before being measured by laser. During the analysis, the fibre bundles are separated by the action of the flow. Thus, a large variation in the thickness distribution can be obtained and values can result in a lower mean fibre diameter. The FMT-Shirley device measures the indirect fineness by examination of the fibre surface by an air flow, while Optical fibre diameter analysis (OFDA) measures the thickness of fibres by the use of a projection microscope and a digital camera *(Beltran et al., 2002; Musio et al., 2018).*

In this work, a system based on scanned image analysis is used *(Müssig and Amaducci, 2018; Schmid et al., 2004).* Fibres bundles were analysed by image scanning and

treated with an image analysis system called Fibershape (IST AG, Switzerland) (Figure 2.8). It determines the average width and thickness distribution of the fibres. The measurement system is based on static image analysis. It automatically detects the fibre bundles, and thanks to their shape and size, it measures their surface area and perimeter. Using a rectangle model, the length and width of the identified objects are described. The system works with a fractal-based algorithm that measures large-scale object thickness, from very coarse fibre bundles to single fibres. It is possible to analyse thousands of objects in a matter of minutes with this system.



Figure 2.8: High-resolution Reflecta scanner connected to Silverfast and Fibershape software.

Scutched, hackled or processed fibres were cut into 20 mm to 50 mm fractions and spread between two glass slides avoiding too many fibres intersect. For each batch, three slides were prepared and inserted into a Reflecta MF5000 scanner. The images were registered with Silverfast software (LaserSoft Imaging, Kiel, Germany) with a 3,200 dpi resolution (Figure 2.9). The images were processed by Fibershape software. A measuring mask saves the parameters and thresholds of the treatment and the algorithm. An adapted standard long fibre measuring mask was used. Calculations were made according to ISO 9276-1 and ISO 9276-2.



Figure 2.9: Preparation of fibres (top) and the scanned image (bottom).

Selection of the Adapted Mask:

The fractal-based algorithm implies an analysis in different steps involving a change in resolution and the measurement of different sizes of fibres. The process starts with low resolution and thick bundles are identified. Then an automatic progressive increase of the resolution measures finer and finer fibres until the 3,200 dpi resolution. A tool enables to check the measured objects at each step. The analysed objects at a specific resolution are presented in green, and turn in black at the following step. Black is then the previous analysed objects and the unanalysed objects. Thus, by observation of the resulted analysis, it is possible to adjust the parameters to the type of material. The parameters of the mask include the measured object thickness, length, ratio, etc.

In our case, we based our analysis on a mask (called “long fibres 8-500um 3200dpi”) developed by IST AG for long fibres such as flax and hemp. First, the detected object thickness was adjusted and maximum thickness was changed from 500 µm to 1,000 µm to take into account the coarsest fibre bundles that are especially present in scutched fibres. The pixel size is between 7 µm and 8 µm.

Secondly, we shifted the minimum analysed length from 25 µm to 1,000 µm in order to avoid the measurement of the dust and to distort the diameters distribution (Figure 2.10).

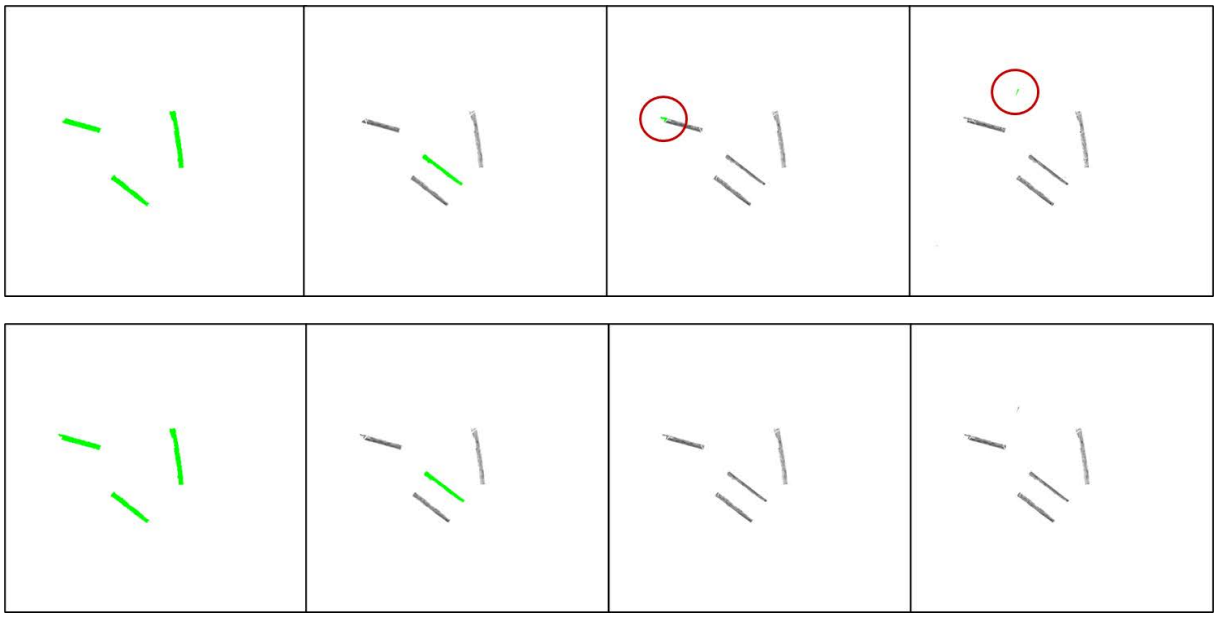


Figure 2.10: Observation of the measured object at four different steps with FibreShape software. A: before modification. B: after modification of the measurement mask.

Finally, the values of four fibres were analysed by FibreShape software using the new mask and were compared with measurement done with optical microscope observations (Figure 2.11). The average thickness of the fibres was similar. (optical microscope: 216.35 µm and FibreShape software: 215.70 µm). Further analysis on more numerous fibres were carried out resulting in very close values.

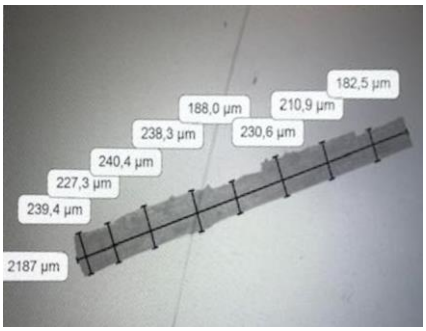


Figure 2.11: Thickness measurement of the 2 mm fibre by optical microscopy.

The method measures the thickness distribution of the fibres. It is important to notice that the fibre bundles have an oval shape. When they are placed on a glass slide, they are generally positioned on their thicker side. This imaging technique will tend to provide enlarged thickness data. As a reference, the Figure 2.12 represents the thickness distribution of textile flax fibres measured by FibreShape.

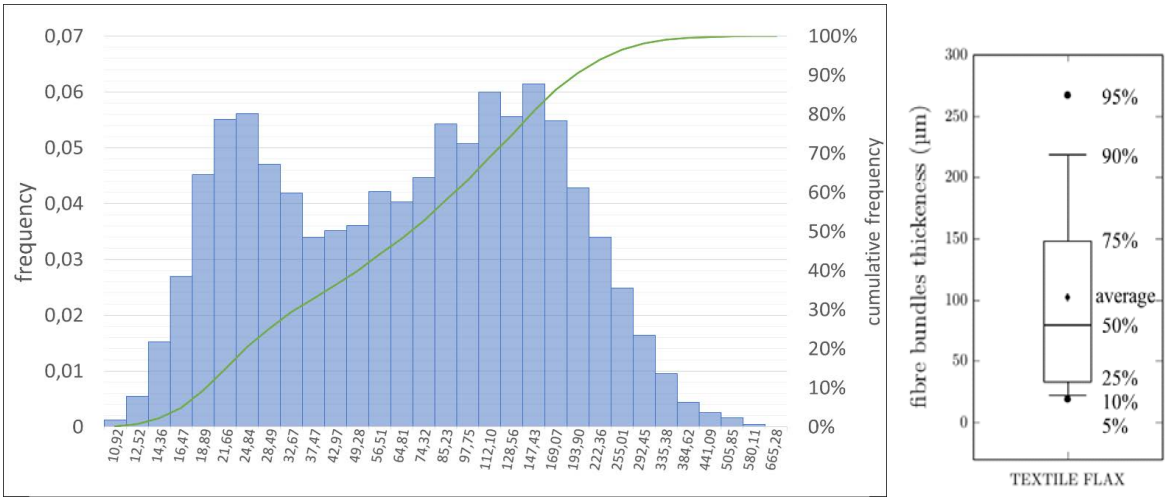


Figure 2.12: Histogram of textile flax fibres thickness distribution of hackled fibres bundles thickness of textile flax and box plot representing the distribution.

The analysis of lab-scutched and hackled textile flax revealed an average thickness of 102 µm, a first quartile of 33 µm, meaning that one quarter of the analysed fibres were thinner than 33 µm, a median equal to 79 µm and a third quartile equal to 148 µm. The results of thickness distribution will further be represented using box plot as presented in Figure 2.12 (right).

Results:

The distribution of fibres bundles thickness was measured on hackled fibres from the three batches. Fibres were extracted and hackled with the Taproot system described earlier. The distribution of Carmagnola hackled fibres thickness is presented in Figure 2.13.

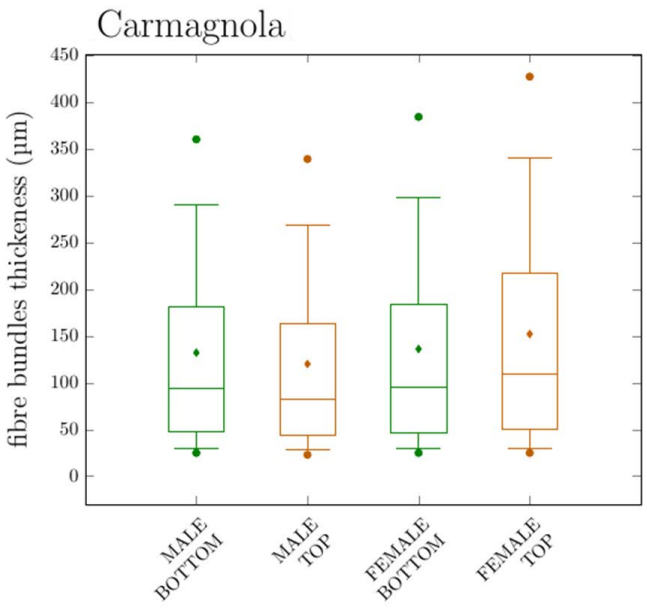


Figure 2.13: Width distribution of hackled fibre bundles in Carmagnola retted plants.

When hemp is grown to senescence, male Carmagnola plants have finer fibre bundles than female plants at the top of the plant. In the lower part of the plant, no difference is observed.

The division potential of the fibres bundles is highly due to the efficiency of retting. It was observed that the retting of Djumbo-20 male plants was less favourable than the female one, due to their dry state. Regarding the thickness distribution presented in Figure 2.14, the female bottom batch contains more refined fibre bundles than the male bottom. In the top part, male and female batches display similar distribution.

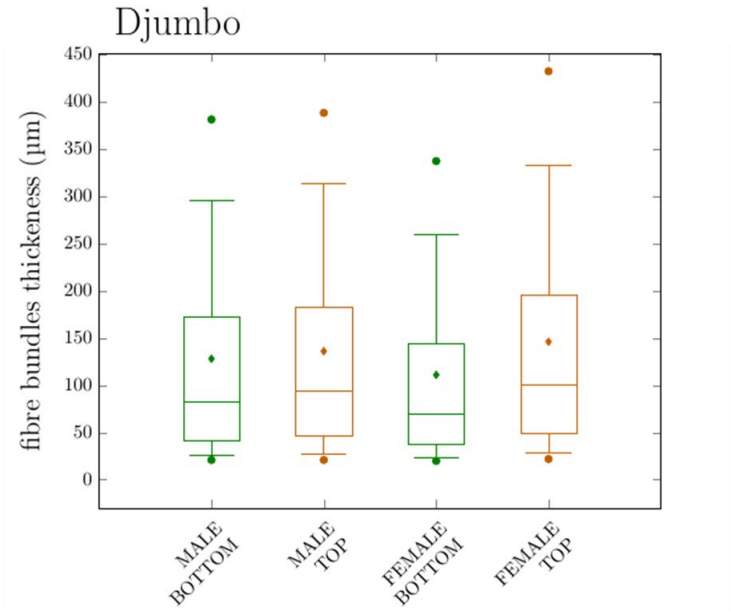


Figure 2.14: Width distribution of hackled fibres bundles from Djumbo-20 retted plants.

Tiborszallasi batch was divided in three portions: top, middle and bottom (Figure 2.15). By comparing the values of the first quartile, median or third quartile, the fibre bundle thickness is decreasing from the bottom to the top. The upper part contains the finest bundles of fibres while the bottom part contains the coarsest bundles.

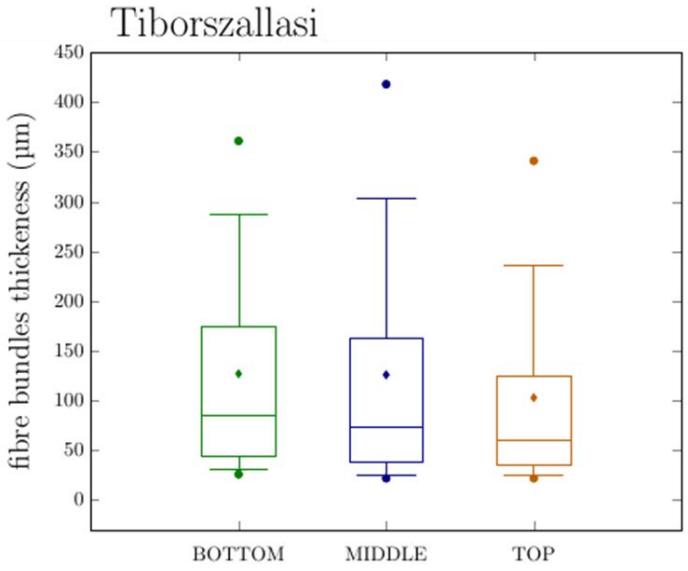


Figure 2.15: Width distribution of hackled fibre bundles in Tiborszallasi plants, in bottom, middle and top portions.

Finally, the median of the batches is between 60 µm and 110 µm and the average is between 100 µm and 160 µm. The values of median and average are closed to the ones of textile flax. However, textile flax contains less thick fibres bundles.

2.2.4. Histologic Analysis

The principle of histological analysis is the optical microscope observation of biological samples. The sample is cut in thin section to let the light going through, and is coloured to observe the several components *(Costa, 2018).* The quality of the image depends, first of all, on the collection of the sample and above all on its preservation. The initial stage, the fixation, is important for the preservation. The sample must be preserved in the best possible conditions before being incorporated into a suitable material to produce sections of a few micrometres. This material is usually paraffin or resin. The latter are hydrophobic and lead to the dehydration of the sample, gradually replacing the water with another liquid, such as ethanol. In addition, for the incorporation medium to well penetrate the tissues of the sample, an intermediate infiltration is necessary, normally using an organic solvent such as xylene or toluene. Once incorporated, the embedding medium get solidified with the sample and are cut into thin sections with a microtome. The rotary microtome is the main tool of the histologist although it is an invention dating back more than a century ago. Sections are placed on a glass slide, coloured, and then observed. The transformation of the sample is summarised in a set of 10 steps:

- Sampling
- Fixation (if storage)
- Dehydration
- Intermediate infiltration
- Inclusion
- Cut
- Rehydration
- Coloration
- Assembly and observation
- Image analysis

Sampling

A portion of (15 ± 3) mm long is cut from the hemp stem with a laboratory knife (Figure 2.16). The sampling area depends on the dedicated analysis, stem nods and surrounding area are avoided. Samples are taken off as delicately as possible in order to maintain all the different elements in their respective places. If the sample is dry, it is first immersed in a large volume of purified water and maintained for 24 h at 40 °C to facilitate further impregnations in the entire tissue.



Figure 2.16: Samples of hemp stems.

Fixation

If the sample needs to be stored for a long period of time before being analysed, the sample can be impregnated with a preservative liquid. Storage in fluid requires the samples to be fixed beforehand, with a fixing solution, then transferred to be stored in a storage solution. Fixation is a chemical process that aims to block and fix the plant tissues as close as possible to their original state. It thus consists in the formation of covalent bonds to link the molecules that form the plant tissues. These solutions are generally composed of one or more diluted aldehydes. The choice of fixative is dependent on the method used, the size and properties of the specimen, and the objectives of the study. The idea is to replace the natural fluid with the fixative. Air in the sample should be avoided, tapping the container can help release bubbles. It is also possible to place the sample for a few minutes under vacuum to promote infiltration. Thicker specimens require more time in solution for fixation to be effective. The specimen must be immersed immediately after collection, respecting the volume ratio (specimen:fixative - 1:20). In addition, fixing is better at low temperatures. A fixed sample sinks in solution and is paler and harder. Insufficient fixation results in a degraded sample while excessive fixation results in a sample too hard and brittle to be sectioned.

The most commonly used fixing solutions are those based on formaldehyde or formalin. Formalin is a mixture of 37% formaldehyde, 10%-15% methanol and 50% water. It is part of the cross-linking fixatives. There is Bouin's liquid (75% picric acid, 20% formalin, 5% acetic acid, water), Davidson's liquid (37.5% alcohol, 25% formalin, 12.5% acetic acid). There are also denaturing fixatives such as Carnoy's liquid (60% ethanol, 30% chloroform, 10% acetic acid), Methacarn's liquid (60% methanol, 30% chloroform, 10% acetic acid). Carnoy's liquid is a fixative recommended for the histological study of fibrous cells and carbohydrates.

After fixation, the specimen should contain as little fixative as possible. It is rinsed with the solvent contained in the fixative. It can for example be immersed in water for several hours in the case of formalin fixation. It is wise to point out that the reintroduction into water can in certain cases break the covalent bonds established and thus re-induce a process of biodegradation. In some cases, it may be advantageous to archive specimens after fixation in a preservation solution. The preservation of the samples is generally done in alcohol and makes it possible to protect the samples from biological agents (bacteria, fungi) and physical destruction for many years. 70% ethanol is one of the oldest preservatives used. Using an ethanol solution that is too concentrated can cause shrinkage. Methanol is also sometimes

used but its penetration capacity is weaker and deep tissues are not well preserved. Isopropanol can also be used but leads to strong discolouration and can cause damage more easily. Glycerol is also a good preservative but has no antifungal properties, it can be a good intermediate in solution with thymol, potassium acetate, distilled water (modified Kaiserling solution). It is important to note that preservative or fixing fluids are not harmless products and certain precautions must be taken when handling them. *(Costa, 2018; Herbin, 2013; Puchtler et al., 1968)*

Infiltration and Inclusion

Paraffin is the most widely used embedding medium in histology. Paraffin is a set of long carbon chains first synthesised in the mid-19th century to make candles. Paraffin is fluid enough when melted which favours embedding and hard enough when it solidifies to allow the sample to be held and easily sectioned. A histological grade paraffin should be used. Samples need firstly to be fully dehydrated before being embedded in paraffin. For this, a series of ethanol baths at different concentrations is carried out. Dehydration is important to for a good impregnation and to preserve the sample. Acetone, methanol and isopropanol can also be used but do not give such good yields. Immersion in 50%, 70%, 95% and absolute ethanol solutions is usually performed. The main idea is to gradually replace the water in the sample with ethanol. The first immersion can be omitted for samples already archived. Paraffin is not soluble in water or in most alcohols, so the sample must be perfectly dehydrated and immersed in an intermediate solvent to make the link between alcohol and paraffin. Toluene and xylene are two hydrophobic organic solvents derived from benzene, they have a strong odour and are miscible in ethanol and in paraffin. Without this infiltration step, the liquid paraffin would be blocked by the ethanol remaining in the sample and the inclusion would only be partial. A similar problem can occur if the samples dry out before infiltration.



Figure 2.17: Samples immersed into alcohol.

Several immersions in the intermediate liquid are necessary, generally three of 10 min to 15 min (Figure 2.17). Between each bath change, it is necessary to remove as much liquid as possible from the sample. Then the sample is immersed in paraffin. The infiltration is generally finished after a whole night or 24 h for the largest samples, but it can remain there for several days or weeks without being altered. Some samples, such as lignified plant samples may require an additional infiltration step such as hot immersion in a 1:1 paraffin - toluene or xylene mixture. The presence of air bubbles can make cutting difficult and should be avoided. Tapping on the edges of glassware helps to counter this problem. Toluene and xylene are aromatic hydrocarbon that can have harmful effects on the body. A less hazardous product, isopropanol, can be substituted to xylene or toluene. In this case, the ethanol series is replaced for an isopropanol series with the same concentrations during dehydration but the immersion times in pure isopropanol is fixed to 1 h. The sample is then placed directly in melted paraffin. Time can be saved because dehydration and intermediate infiltration are carried out in the

same solvent. In this case, the period of infiltration in liquid paraffin is recommended longer (48 h). Another substitute to toluene and xylene is commercial products such as Histoclear, limonene based. *(Gonçalves et al., 2018; Herbin, 2013; Reeve, 1954; Viktorov and Proshin, 2003).* After impregnation, the sample is placed in a mould (about 2 cm^3) and is completely covered with new liquid paraffin. The sample is placed in the middle of the mould and carefully adjusted and aligned with respect to the stem axis so that it is straight for cutting sections. The paraffin solidifies quickly, so the operation must be done quickly and gently. The block is cooled down for at least 6 h at ambient temperature. The sample can be stored for several month in this state.

Microtome Cutting

The samples within the paraffin blocks are then cut with a rotary microtome. The first step is to trim the block to a regular, uniform trapezoidal shape before placing it on the microtome. Large sections are first cut to remove the excess of paraffin and reach the sample. Then sections of about 9 µm thick are cut. Thicker sections cause too much background noise, too much staining and out of focus during microscopic analysis. Thin sections generally provide clearer and cleaner images but are harder to make and to colour. The cutting angle of the microtome must be adjusted so as not to generate great friction and not to deform the sample. Generally, better cuts are obtained with a high cutting speed. Ribbons of sections are obtained following the cut (Figure 2.18). Many sections are advised for more representativeness. The ribbons are placed on a slide covered with an adhesive, such as albumen, and left to dry. *(Clair et al., 2005)*



Figure 2.18: Samples inside a block of paraffin. Cutting the edge of the block. Microsection ribbon done by microtome.

Staining

Most dyes are hydrophobic, so two steps are necessary before staining: deparaffinization and rehydration. The first step is done with a solvent to dissolve the paraffin, the same as for infiltration (xylene or toluene). The second step is done by a series of regressive concentration of alcohol (usually the same as for infiltration: ethanol) down to distilled water. Dewaxing can also be done with isopropanol. In this case, the slides are heated to 60°C-70°C until the paraffin is melted and then immediately immersed in a 100% isopropanol solution. This step is repeated until the slide is paraffin free. Then the slide is rehydrated using a series of isopropanol baths with decreasing concentrations. The slide is immersed into deionised water and into bathes of dyes (Figure 2.19). After being rinsed, one drop of deionised water is added on the slide and a coverslip is placed on top. Slides can be sealed with nail polish for longer preservation of hydrated specimens. The mount is then observed by optical microscope Olympus PMG3 (Japan).

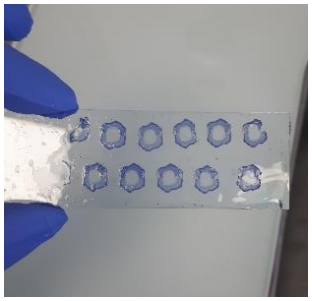


Figure 2.19: Cross-sections on a slide after dying.

The staining procedure helps to distinguish the different components. It involves the use of dyes that absorb light at different wavelengths. Staining intensity is directly related to dye concentration and staining time. Staining can occur by adsorption at the surface of tissues. A dye makes possible the binding to the tissue and provides the colour. Dyes used to colour the surface of cellulosic and fibrous materials generally have ionic charges, either cationic or anionic. In general, cationic chromophores tend to adsorb more on cellulosic materials because they have rather negatively charged surfaces *(Alturkistani et al., 2016; Hubbe et al., 2019).*

Several dyes can be used for differentiating between components in cell walls. With bright light microscopy, a common staining is the double dying of alum carmine that stains cellulose light red and iodine (or brilliant) green that stains lignin green. Another dye usually used is toluidine blue. It has a metachromasia property and is able to change their colour when bound to the tissue compared with its colour in solution. It happens when the light absorption of the dye changes after the binding to the tissular components. Toluidine blue stains the carboxylated polysaccharides pink-red and the lignin bright blue-green. Thus, the xylem appears green or blue-green, the phloem red, sclerenchyma blue-green and collenchyma and parenchyma cells red-purple. Other dyes such as Congo red stains red the crystalline cellulose and phloroglucynol-HCl detect lignin in violet-red colour and hemicellulose in cherry red colour.

It is also possible dyes to perform fluorescence observations. The fluorochrome is a fluorescent chemical compound that absorbs light energy of a specific wavelength and re-emits light at a longer wavelength. Calcofluor white stains cellulose blue. Acridine orange is metachromatic dye that makes it possible to enhance the contrasts of the parietal network. It reveals cellulose green, lignin red and when both compounds are present in large quantities it stands out in yellow, as it is the case in the middle lamellae *(Abot, 2010; Blake et al., 2008; Bonatti et al., 2004; Crônier et al., 2005; Liu et al., 2015a; Marrot et al., 2013; Schäfer and Honermeier, 2006; Snegireva et al., 2015).*

Immunohistochemistry can also be used to differentiate between component plant cell walls. It involves appropriately labelled antibodies binding specifically to their target antigens in biological tissues *(Behr et al., 2019; Bourquin et al., 2002; T. E. Chernova et al., 2018; Gorshkova and Morvan, 2006b; Gorshkova et al., 2004).*

In summary (scheme in Annex 2):

1- Taking off 10 mm to 15 mm long sample
2- Fixation in Carnoy's liquid for at least 2 hours
3- Washing and preserve in 70% ethanol
4- Dehydration and infiltration in isopropanol series 70%, 95%, 99% (one hour each)
5- Inclusion in paraffin (one night)
6- Block, cooling down (6 hours) and sectioning
7- Dewax and rehydration in isopropanol series 99% to 70% and distilled water

8- Staining
9- Montage and observation (Figure 2.20)

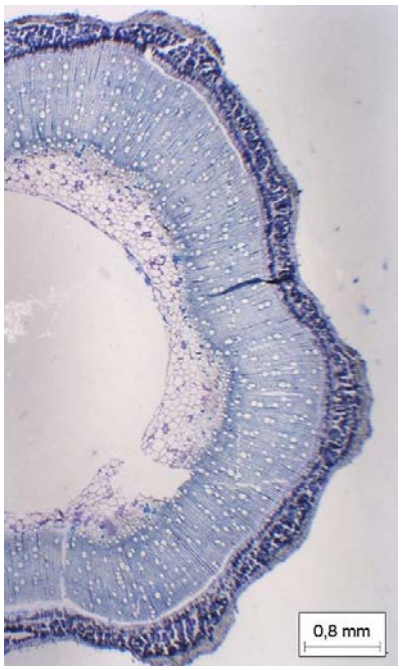


Figure 2.20: Half cross-section of hemp stem stained with toluidine blue.

Results:

The analysis of the morphology of the transverse section of the fibres was done on one batch: Carmagnola male. The images of sections from the bottom, middle and top part are presented in Figure 2.21. Qualitative observations were made on those samples.

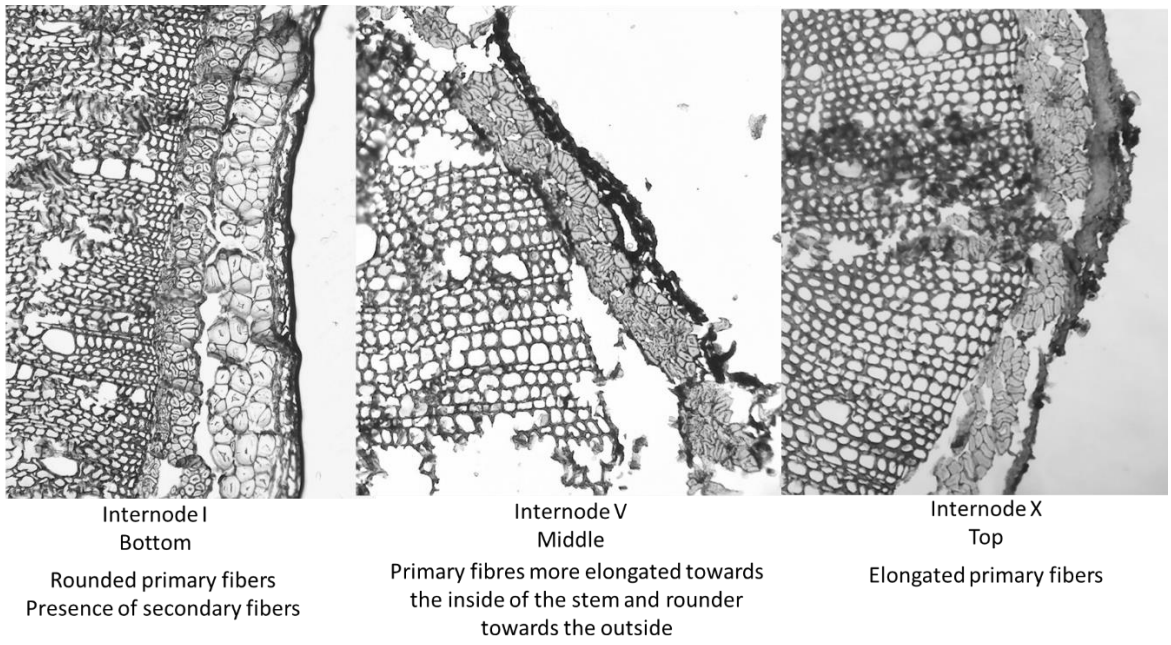


Figure 2.21: Images of cross-sections of a male Carmagnola hemp stem taken from bottom, middle and top parts of the stem.

The shape of the fibres might be different from the bottom to the top. They are more elongated at the top and more rounded at the bottom. In the bottom part, secondary fibres are present. Fibres either from the top, middle or bottom part seems to be filled.

2.2.5. Discussions

According to the literature, there is evidence of genotypic variation in fibre properties, but due to the great variability in fibre characteristics caused by the environment and the large influence of fibre characterisation methods, the identification of varieties with specific fibre qualities is very difficult *(Amaducci et al., 2015; Berenji et al., 2013; Cromack, 1998; Zatta et al., 2012).* The fibres evolve with plant development and the observed tendencies in fibre properties might differ between flowering and seed maturity. In this study, depending on the variety and the harvesting time, fibre properties between male and female or top and bottom part of the stem did not reveal the same tendencies *(Berenji et al., 2013; Amaducci et al., 2015).* Around the onset of flowering, the flow of nutrients is shifting toward to the development of flowers and seeds, and less to the development of stems, leaves, and roots. The flowering time of male and female happens at distinct moment, then the development of fibres is impacted.

When harvested at seed maturity and processed by scutching and hackling systems, the bottom part of Carmagnola variety revealed a higher fibre content than the top part, and the male plants shows a slightly higher fibre content than female plants. These findings support results from literature through histological approach *(Cerri et al., 2022)*. The male top batch has finer fibre bundles and female top batch has the thickest, however male and female bottom batches display a similar distribution of fibres bundles thickness. A significant difference in tensile modulus is observed between fibres from male and female bottom parts. This is in adequation with studies from the literature that reported that the fibres of female plants have thicker cell wall at seed maturity than the fibres of male plants, which would imply that their lumen would be smaller and their tensile properties higher *(Cerri et al., 2022)*. Mechanical properties from the top and the bottom parts of male plants revealed very close properties. The cross-sections seem to confirm the results. The fibres seem to be full either at the top or bottom of the male stem, which is in agreement with tensile tests with the assumption that the lumen size influences the tensile strength and tensile modulus *(Richely et al., 2022)*. Moreover, the fibres from the top appear smaller than the ones from the bottom and could form finer fibres bundles, revealed by the fineness measurement.

At full flowering stage, different patterns are observed. Considering the Djumbo-20 variety grown in 2021, alike fibre content are observed between male and female, and top and bottom, as well as similar mechanical properties. However, the same variety grown one year later and also harvested at full flowering generated a higher fibre content for the male batch than the female batch. A difference can be observed in fibre bundles width distribution between the female batches: the female top batch reveals the finest fibres. Considering the Tiborszallasi variety, more heterogeneity can be observed in the fibre properties from the top and the bottom. The bottom fibres reveal higher mechanical properties and coarser fibres than the top while a higher fibre content was obtained for the top part but contained much residue. The bottom fibres might reveal a more advanced maturation stage, as it was described by Amaducci et al. (2005); maturation is acting from the bottom to the top *(Amaducci et al., 2005).*

A deeper study on the dioecious and monoecious variety is performed in the next section. Varieties grown in the same conditions were studied and the evolution of the fibres properties along with the plant development investigated.

2.3. Evolution of Hemp Fibre Properties During Plant Development

2.3.1. Experimental and Design Plan and Analysis

The properties of fibres evolve with the plant's growth. They are generally considered optimal at flowering time when hemp harvesting for fibres is targeted. It is assumed that the primary fibres are rather filled without much appearance of secondary fibres. However, no real screening has been carried out to assess whether some varieties give a better balance than others. The objective of this work is to evaluate the influence of harvesting time on fibre characteristics by comparing several varieties. The filling rate of fibres, the fibre yields, the tensile properties of fibres, the technical fibre thickness and the presence of secondary fibres are observed. These data will be used to determine the optimum harvesting period to expect high quality textile fibres. Varieties are described according to their estimated flowering time in Angers (France) and are divided in two categories: early-flowering (when full-female flowering is estimated before the 10th of August) and late-flowering (when full-female flowering is estimated after the 10th of August). Hemp plants were grown over three consecutive years, 2021, 2022 and 2023. The five studied varieties are presented in the Table 2.5.

*Table 2.5: The studied varieties. *V4 was studied in 2021 and 2022, and V5 was studied in 2022 and 2023.*

Variety	Commercial name	Gender	Group of genotypes	Estimated female full-flowering date
V1	Djumbo-20	Dioecious	Early flowering	August 8 / (July 7: males)
V2	USO-31	Monoecious	Early flowering	July 28
V3	Carmagnola	Dioecious	Late-flowering	August 15
V4*	Dioïca-88	Dioecious	Late-flowering	September 9
V5*	Muka-76	Monoecious	Late-flowering	August 21

The seeds of the V1, V2, V4 and V5 varieties were provided by Hemp-it company (Hemp-it, France) and those of V3 are from Assocanapa Associazione (Vila Morello, Italy). All the varieties were sown by Henri Pomikal on the 12th of May 2021, on the 14th of May 2022 and on the 19th of May 2023 with a sowing density of approximately 500 units per m². The field trials took place in Martragny on the Plain of Caen, Normandy, France (49° 15′ 4.739 N– 0° 35′ 48.80″ O) (Figure 2.22).



Figure 2.22: Field trial in Martragny during the harvest.

Samples were realised at five dates prior harvesting in 2021 and three dates in 2022 and 2023 (Figure 2.23): on July 2 (52 days after sowing), July 13 (D63), July 21 (D71), August 4 (D85) and August 17 (D98); three dates in 2022: June 30 (D48), July 18 (D66) and August 3 (D82); and one date in 2023: August 2 (D76).

Samples are (15 ± 3) mm long pieces of the stems. In 2021, samples were collected in the bottom, middle, and top parts of the stems whereas in 2022 and 2023, they have been collected only from the bottom part. By measuring the total bast fibre content, it was found that the section located between 30% and 40% of the height of the plant is the most representative of the entire stem *(van der Werf et al., 1994)*. Samples from the bottom part were collected at about a quarter of the plant height, samples from the middle, at half-height and samples from the top, at the third quarter.

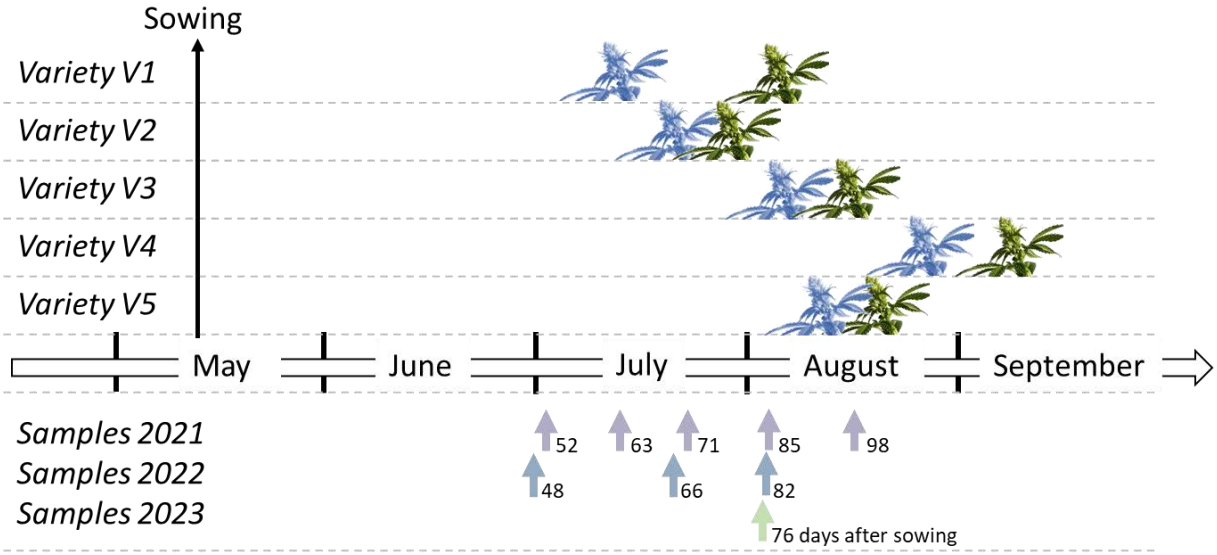


Figure 2.23: Time of the predicted male full-flowering (blue) and female full-flowering time (green) for each variety, and the dates of the samples for each year with the number of days after sowing.

Three replicates for each sampling were studied. The histological procedure was carried out as described in section 2.2.4. The image analysis is described below.

Image Analysis

Observed images were analysed with Fiji software (ImageJ, 2021). Different components of the stem can be observed in the transverse section, such as the xylem, the phloem, the primary fibres, the secondary fibres, the cell walls, the lumen and the epidermis as illustrated in Figure 2.24. The diameter of the stem section, the radial length of the primary and secondary fibre layers, the large diameter and small diameter of the primary fibre cell and the cell wall thickness are measured using the line selection tool. The area of the xylem, the phloem, the primary fibres and the area of the lumen are measured using the freehand shape area selection tool. DOI are registered. Data are transferred into an Excel document for data treatment.

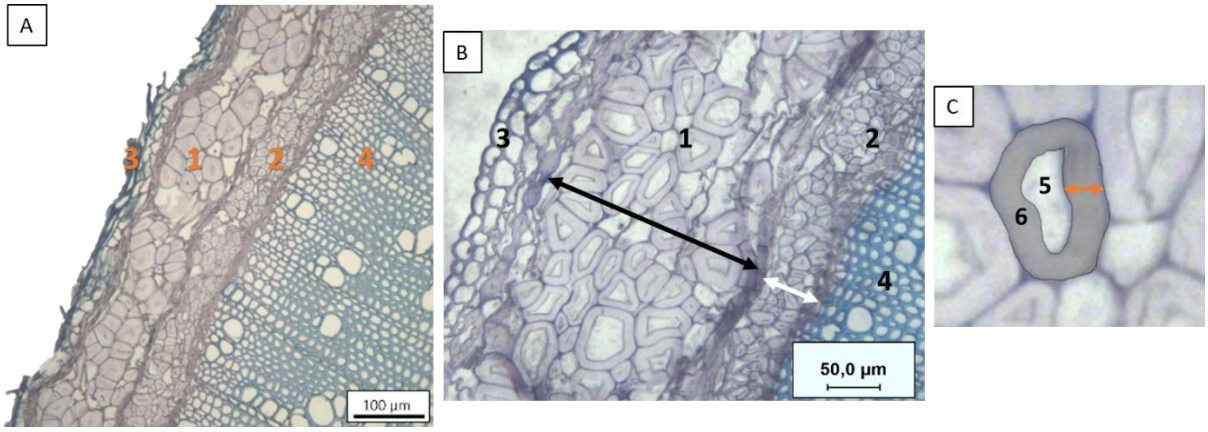


Figure 2.24: Transversal sections of hemp stem with gradual observation size from A to C. Sections are colored with carmine alum and iodine green. 1: primary fibres; 2: secondary fibres; 3: epidermis and cortex; 4: xylem; 5: lumen; 6 (dark grey area): cell wall. Black line: primary fibres layer thickness. White line: secondary fibres layer thickness. Orange line: cell wall thickness.

To optimise the segmentation and carry out the analysis more quickly, machine-learning can be a very powerful tool *(Laine et al., 2021)*. In this work, an automating machine learning segmentation tool on Fiji software and with Matlab software were tested. The Fiji tool was not able to differentiate the cell correctly (no image shown). The program created in Matlab consists in applying a field correction, choosing the area of interest and optimising the contrast on this area, binarising the image and detecting the contours (Figure 2.25). However, this process was not able to bring out the desired analysis. Further works need to be done to create an adapted process or to develop a suitable deep learning algorithms for this type of image analysis.

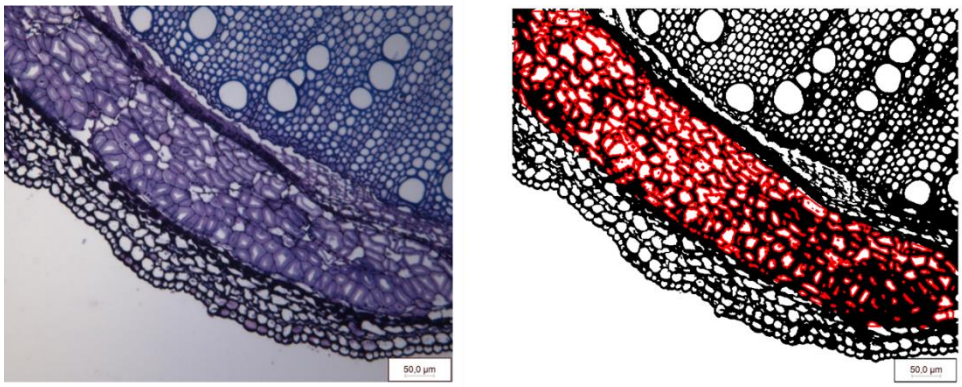


Figure 2.25: Process of fibre analysis using Matlab.

In this work, measurements are taken manually with Fiji software in each third of the section whenever possible. The area of the primary fibre layer (A_{PF}) was calculated using the diameter of the stem and the average radial thickness of the layer, following the equation (2-1). For the calculation, the layer is assumed to be regular along the section.

$$A_{PF} = \pi \times \left(\frac{d_{stem}}{2} - th_{Ep}\right)^2 - \pi \times \left(\frac{d_{stem}}{2} - th_{Ep} - th_{PF}\right)^2 \quad (2\text{-}1)$$

Where d_{stem} is the diameter of the stem, th_{Ep} and th_{PF} are the thickness of the epidermis and the primary fibre layer, respectively.

The area of the secondary fibre layer was calculated in the same way and the area of the total fibres area (A_{TF}) is the sum of the two areas. The proportion of secondary fibres inside the stem ($\%_{SF}$) is then calculated according to the equation (2-2).

$$\%_{SF} = \frac{A_{TF} - A_{PF}}{A_{TF}} \times 100 \quad (2\text{-}2)$$

The maturity of the fibres is calculated by the filling rate of the fibres, i.e., by the area of the cell wall (A_{CW}) in relation to the area of the cell (A_C). The filling rate ($FR(\%)$) is calculated by the equation (2-3). At least 50 fibres are measured for each batch.

$$FR(\%) = \frac{A_{CW}}{A_C} \times 100 \quad (2\text{-}3)$$

2.3.2. Fibre Development Monitoring

2.3.2.1. Evolution of Hemp Plants

Properties of the fibres can be affected by the development of the plants. The growth of the plant is depending on the climatic conditions, then weather data was collected in a meteorology station placed in the field. From sowing to harvesting dates, the field received a total precipitation of 166 mm in 2021 (98 days after sowing), 118 mm in 2022 (82 days after sowing) and 144 mm in 2023 (83 days after sowing), and an average temperature of 16.2 °C,17.1 °C and 16.8 °C respectively in 2021, 2022 and 2023. Prediction of plant growth stages is sometimes done by use of the cumulative temperatures received by the plants after sowing. The stages are determined using the cumulative growing degree-day (GDD_n) *(Rahemi et al., 2021).* Growth degree days (GDD) were calculated considering a base temperature of 1°C, indicating the biological temperature minimum of the beginning or end of growth and using the equation (2-4) *(Amaducci et al., 2005; Goudenhooft, 2018).* For flax, fibres are considered mature when the cumulative GDD reaches from 950 °C.day to 1,100 °C.day *(Goudenhooft, 2018).*

$$GDD_n = \sum_{i=1}^{n} \frac{T_{max,i} + T_{min,i}}{2} - T_{base} \quad (2\text{-}4)$$

At harvest, the GDD_n was higher in 2022. They valued 1,165 °C.day, 1,208 °C.day and 1,191 °C.day, respectively in 2021, 2022 and 2023. Weather data from 2021 are presented in Figure 2.26 and weather data for 2022 and 2023 are presented in Annex 1.

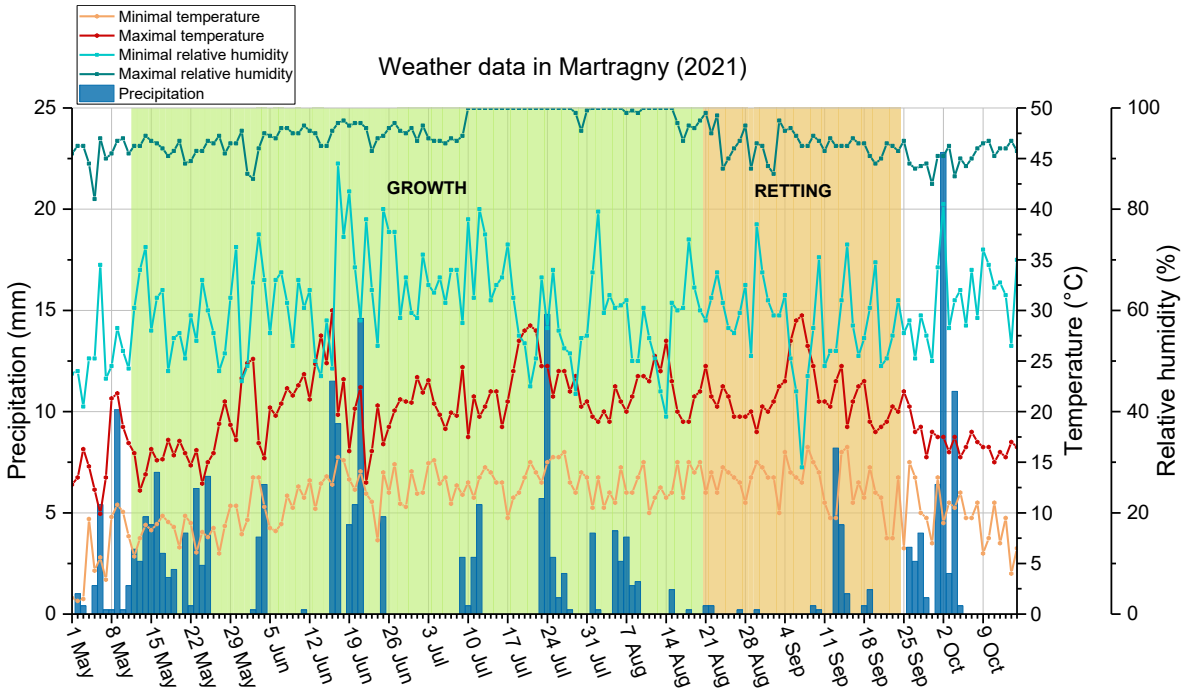


Figure 2.26: Weather data in the field trial in Martragny during hemp growth cycle in 2021.

The nitrogen residue was evaluated on the 0.60 m soil top layer to 66 kg N/ha in 2021 and 89 kg N/ha in 2023 (data were not available in 2022). The average plant density of the different varieties was approximately 255 plants/m^2 for both years.

The height and the thickness of the stems are used to define the morphology of the plants. The evolution of plant size in terms of height and diameters in 2021, 2022 and 2023 are represented in the Figure 2.27. The plants are slightly smaller in 2021 than in 2022 and 2023 despite more rain. The value of the GDD_n was smaller in 2021 than 2022 and 2023. In 2023 the nitrogen input was higher than in 2021.

According to Van der Werf (1994), plants with a longer vegetative phase tends to grow taller and thicker *(van der Werf, 1994).* In this work, at the last samplings of 2021 and 2022, the early-flowering variety V1 tends to have thinner and shorter stems and the late-flowering varieties V3 & V4 tend to have bigger and higher stems.

Amaducci et al. (2005) described that the maturity of the fibres is dependent on the size of the plant, the value of the internode and indirectly to the plant density (stem diameter) *(Amaducci, Pelatti and Bonatti 2005)*. They found that the bigger the stems, the higher the filling value. In this work, no distinct pattern could be revealed between filling rate of primary fibres and the morphology of the stem. Likewise, no clear relation between secondary fibre proportion and stem morphology can be defined. It is, however, noticed that stem of less than 170 cm high and 6 mm thick exhibit secondary fibres in the middle part of its stem. It was reported that the secondary fibres front high rises with the dry stem weight and the size of the plant *(Westerhuis 2019).*

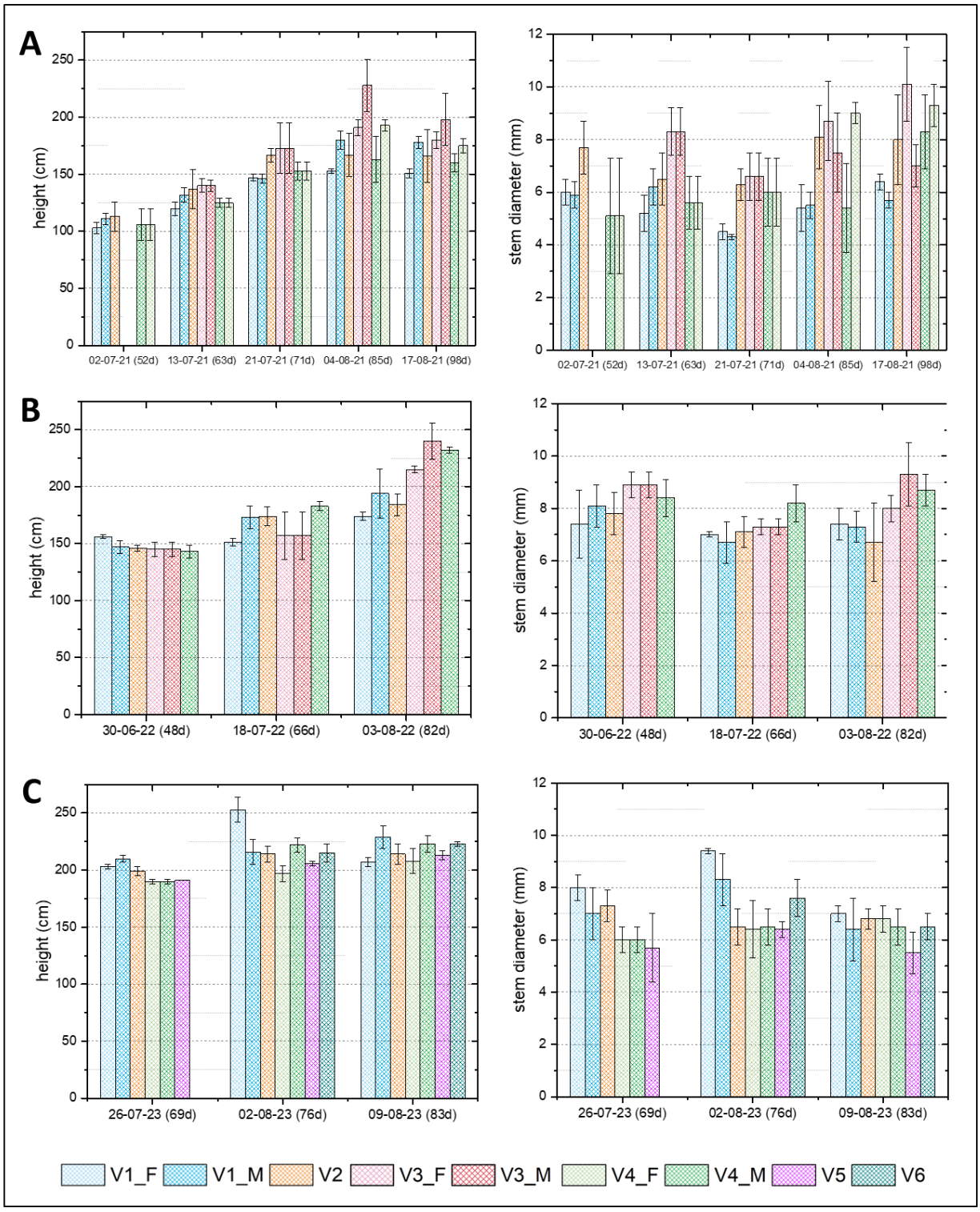


Figure 2.27: Evolution of the stem height (left) and stem diameter (right) in 2021 (A), 2022 (B), and 2023 (C). F: Female plants, M: Male plants. In 2022 (B) gender of V4_F and V4_M are indistinguishable, the absence of flowers prevents the plants from being recognized.

2.3.2.2. Evolution of Bast Fibres Formation During Growth

Maturity of Primary Fibres

Fibres maturity was estimated through the evolution of the thickness of the primary fibres cell walls measurement along the period. It was observed that the cell wall thickness increases during both vegetative and flowering phases. Cell wall filling reaches 75% and 83% on average in late July, respectively in 2021 and 2022 and 84% and 85% at the beginning of August (89% in 2023). The filling of the fibres over time in 2021 and 2022 presented on the Figure 2.28.

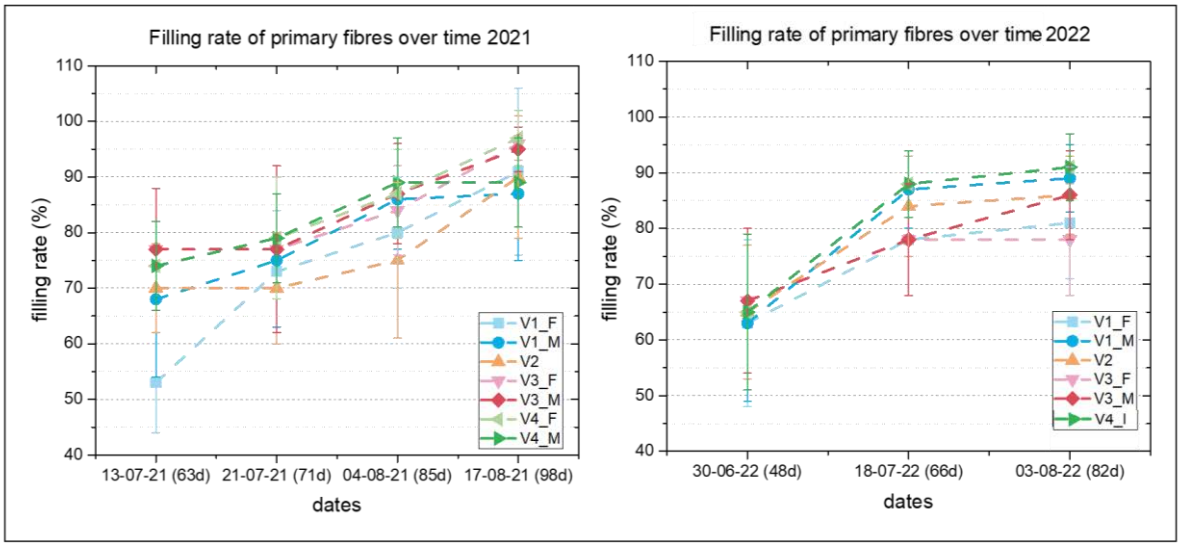


Figure 2.28: Filling rate of primary fibres during 2021 and 2022. F: Female plants, M: Male plants, I: Indistinguishable gender, the absence of flowers prevents the plants from being recognised.

Varieties filling rates show distinct patterns. The filling rate is not related to the flowering time of the variety. For example, the varieties V3 and V4 did not start flowering in mid-July and primary fibres are already filled at a rate higher than 75%. If one considers mature fibres the cells with a filling rate higher than 65% as described in *(Amaducci et al., 2008b)*, these results show that fibres are mature much before flowering.

The latest-flowering varieties show an early increase in the filling rate compared to early-flowering varieties. In 2021 and 2022, this tendency is well observed along the growth with the variety V4 (the latest flowering one) showing the highest filling rates, however, without large differences. In 2021, late-flowering variety V3 also shows a high filling rate compared to the early-flowering ones, V1 and V2, and in 2022 its rate was around the lowest values of the early-flowering ones.

Male and female filling rates show distinct behaviours. Fibres from male plants reach a faster increase of degree of filling than the ones from female plants. Only the last sampling in 2021 revealed a lower filling rate for male plants. Cerri et al. reported that female plants tend to reach a higher filling rate than male plants at seed maturity *(Cerri et al., 2022)*. It looks like growing flowers compete with maturing fibres in a sense that the energy consumed for developing flowers and seeds represent a shortage for filling fibres.

Secondary Fibres

Secondary fibres were identified in the bottom, middle, and top part of the stem in 2021 and only in the bottom part in 2022. The proportion of secondary fibres respectively to the total bast fibres is presented in Figure 2.29 for each year.

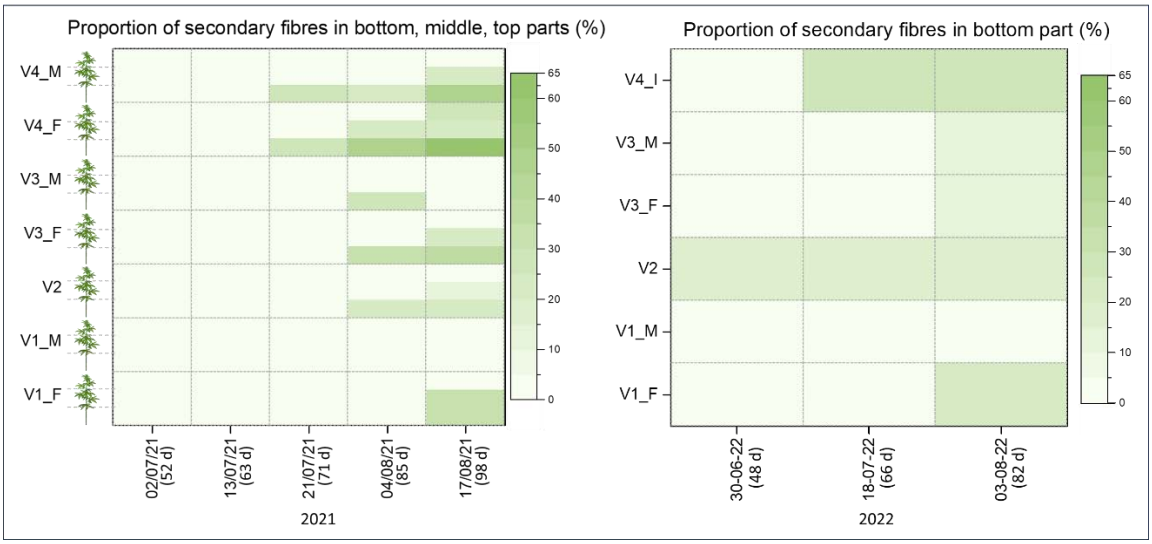


Figure 2.29: Representation of the ratio of secondary fibres to total bast fibres in bottom, middle and top part of stems in 2021 (A) and in bottom part in 2022 (B). F: Female plants, M: Male plants, I: Indistinguishable gender, the absence of flowers prevents the plants from being recognised.

Secondary fibres appeared first in the bottom part of the stem in late July in 2021 and at the end of June in 2022. They appeared in low proportion in relation to total bast fibres. The secondary fibres were present in different proportions depending on the variety. They were observed in a higher proportion in variety V4 both years. The proportion of secondary fibres was higher than 20% of the bast fibres from the second part of July, corresponding to 63 days (2021) or 66 days (2022) after sowing, and came to more than 50% in mid-August. They reached the top part of the stem of the variety V4 on the last sampling, August 17, 2021.

For the same variety, the male and female plant did not have the same pattern. Secondary fibres appeared in higher proportion in female plants [in accordance with *(Cerri et al. 2022; Mediavilla, Leupin and Keller 2001)*]. However, no significant differences could be determined. Van Der Werf et al. established a relation between the appearance of secondary fibre and the mass of the dry plant, it would be interesting to compare the secondary fibre proportion with the weight of the fresh plant.

2.3.2.3. Growing Late-Flowering Varieties

The time of flowering is considered as a crucial indicator for the time of harvesting hemp for fibres. It usually corresponds to the moment when primary fibres are filled, lignification is low, and fibre yields is the highest. In some areas, in middle latitude where too humid weather is occurring at the end of the summer, it was not worth considering growing late-flowering varieties as flowering would happen too late and the fibres were supposed not to be filled enough to be harvested several weeks before flowering. During the development of the fibres, the cell walls thicken from the outside to the inside and the content of cellulose increases. Large cell walls are indicative of a mature fibre. Filled fibres weigh more than hollow fibres and have higher mechanical properties. Hence, the fibre yield, which is measured according to the extracted long fibre weight and the harvested weight per hectare, is enhanced with heavy fibres, and during the extraction of long fibres, fibres with high enough mechanical properties

do not break. The properties of the fibres are also directly influenced by the genotypes. Hence, having a deeper look on the state of the fibres for different varieties through their growth would favour cultivars selection optimisation.

It appears that primary fibres may be highly filled before the flowering time. This is not in agreement with the suggestion from *(Mediavilla et al. 2001)* who reported that fibres are getting mature at the flowering time. It also appears that the late-flowering variety tends to have their primary fibres filled earlier in the growth plant cycle with higher rates than the early flowering varieties (Figure 2.28). Genotype influences fibre maturity. One explanation could be that the early varieties allocate less energy to develop their fibres and their stem, because the plant needs energy for the reproduction organs development at an advance stage than later flowering varieties.

On another side, it was reported that late-flowering varieties tends to grow faster, higher, with bigger stems and secondary fibres appear to support the stem weight and resist against climatic events *(van der Werf 1994; Westerhuis et al. 2019).* Those secondary fibres are unwanted for textile applications, but they are expected to be separated from the long fibres during the scutching process. They can constitute a large part of the scutching tows that can be valorised in technical applications. In this work, secondary fibres tend to appear in higher quantities in the late variety V4 than in the other varieties. While the earliest-flowering variety V1 has the least secondary fibres and a lower filing rate, the latest-flowering variety V4 has the most secondary fibres and the higher filing rate. Nevertheless, secondary fibres only reached the bottom of the stem before August and in proportion lower than 30%.

The full cross-sections and the block of hemp stem included into the paraffin were also observed using an optical microscope. Images are presented in Figure 2.30. The stem diameter and the xylem area were measured. The solid part was treated with Paint 3D and measurements were done with Fiji software.

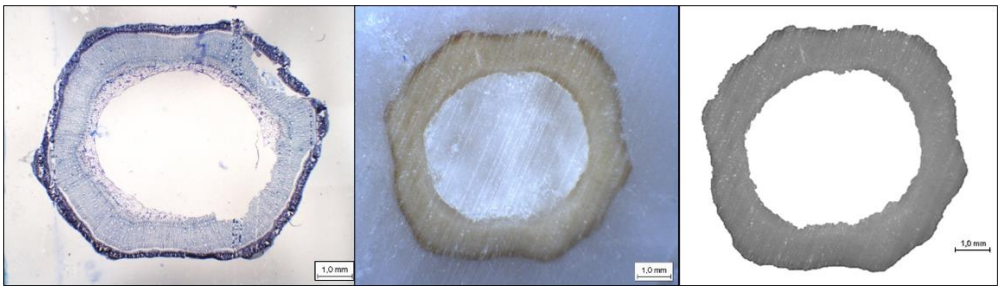


Figure 2.30: Transverse views of hemp stems. From left to right: stained transverse section after micro-cutting, transverse section of hemp stem inside a paraffin block, image treated with Paint 3D.

The transverse sections of the stems are broadly described in area of xylem, phloem which contains the bast fibres, epidermis and pith. The distribution for each variety from the last samplings in 2022, is illustrated in Figure 2.31.

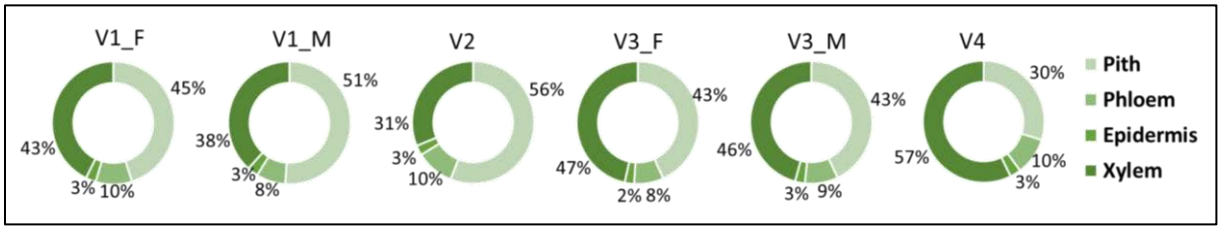


Figure 2.31: Element distribution in volume in a transverse view of hemp stems according to varieties.

We observed that the early varieties (on the left) are composed of a smaller part of xylem compared to the late flowering (on the right). Hernandez et al. reported that more secondary fibres were observed when the xylem layer was thicker *(Hernandez et al., 2007a).* In our case, the proportion of secondary fibres was higher with variety V4 and lower in the variety V1_M, which is coherent with work from literature.

2.3.2.4. Harvesting Before Flowering

For the long fibres extraction, the plants are processed by a scutching machine that requires the stems to be 80 cm to 1 m-long. The hemp plant reaches around two metres high. During harvesting, the stems are cut into two parts of about one metre each. The optimal targeted stem height would be more than two metres to optimise biomass yield and usable length. The stem height is continuously increasing during the plants development and seems to be stable from the beginning of August, reaching heights between 150 cm to 260 cm depending on the variety and the year.

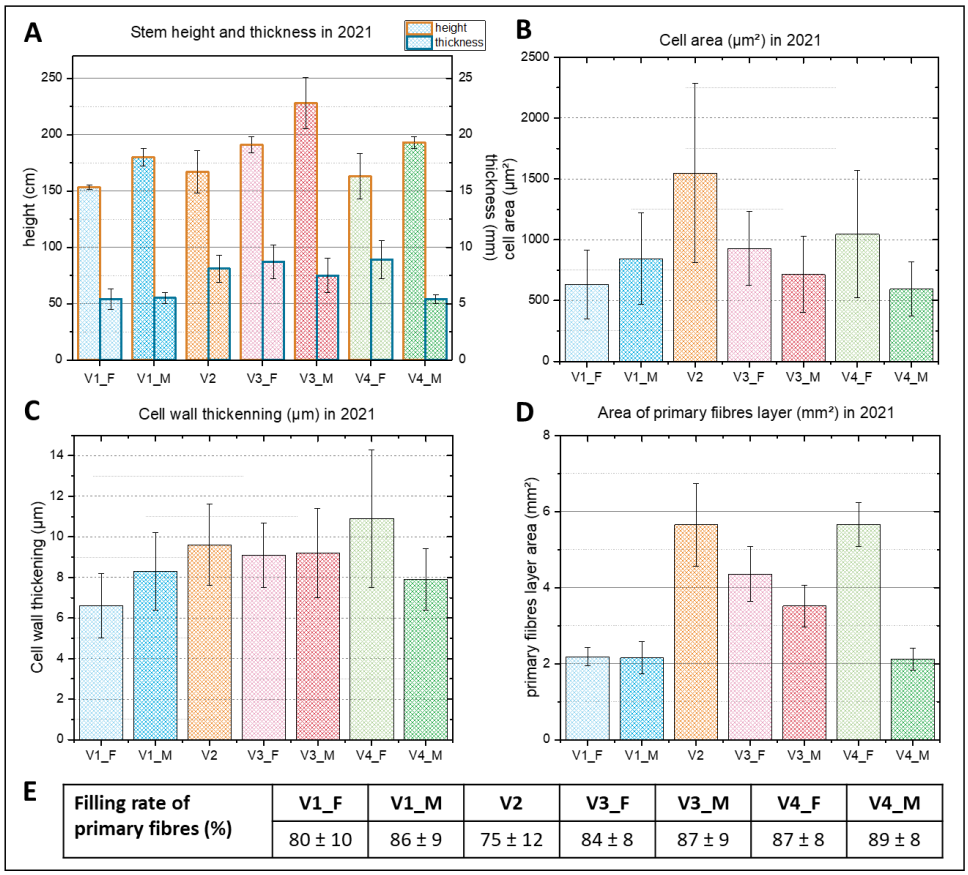
Considering the stem height, the filing rate of primary fibres and the presence of secondary fibres in the studied plants from 2021, a time of harvest was suggested at the end of July or in early August in 2022 and 2023. Samplings were done at the beginning of August in 2021, 2022 and 2023, corresponding to 85, 82 and 76 days after sowing respectively. Each year, the filing rate of primary fibres was higher than 75% for all varieties, and up to more than 86% for the late-flowering variety V4. The presence of secondary fibres was limited and reached the middle of the stem for the female plant of V4 variety (in 2021) only. It was reported by Amaducci *et al.* (2005) that high filling of primary fibres is observed sooner at lower internodes up to the beginning of flowering and the degree of filling was similar along the stem at full flowering *(Amaducci et al. 2005)*. In this work, samples were also taken in 2021 from the middle and top parts of the stems, and from the beginning of August, the plants in all three sections had a filling rate greater than 75%.

The morphologies of the fibres were more deeply compared among varieties at this period and additional measurements were carried out and results from 2021 and 2022 are exposed in Figure 2.32. At that time, V1 is at full flowering (code 2103; 2203 according to Mediavilla 1998), V2 is flowering (code 2304), V3 is at the beginning of flowering (code 2102; 2201) while V4 has not started flowering or is at the early beginning (code 2100; 2200).

The cell area measurement corresponds to the mean transverse area of a single fibre. Thin fibres are sought after for textile applications in order to produce fine and strong yarns. The minimum size of a yarn is limited by the minimum number of fibres per cross-section, which is around 100 fibres per cross-section depending on the application and the type of spinning. Fine fibres reduce a limit size. A stronger yarn can also be spun by increasing the number of fibres in a cross-section. The number of fibres can be increased with fine fibres to reach a defined size of yarn. Results display a higher cell area for the monoecious plant (V2) than the dioecious ones. Large variations in cell area are observed resulting in heterogenous size of the fibres.

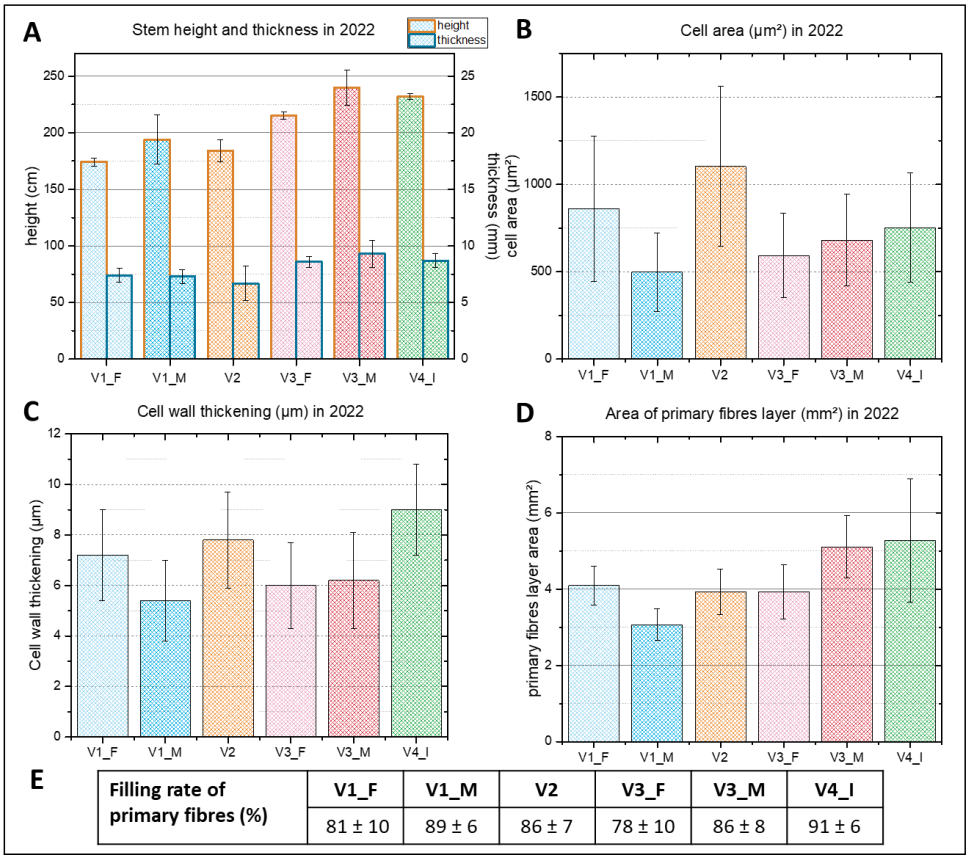
Cell wall thickening corresponds to the increase in the S2-layer of the fibres and is related to the filling rate. The cell wall thickening confirms the fact that maturation at harvest time is more completed for the late-flowering variety V4 than the other varieties.

The area of primary fibres gives information about the content of fibres inside the stem. A large primary fibre layer would correspond to a higher proportion of fibres and then might lead to a higher long fibre yield. It is observed over the two years that the variety V1 has the thinnest primary layer area compared to the other varieties.



E

Filling rate of primary fibres (%)	V1_F	V1_M	V2	V3_F	V3_M	V4_F	V4_M
	80 ± 10	86 ± 9	75 ± 12	84 ± 8	87 ± 9	87 ± 8	89 ± 8



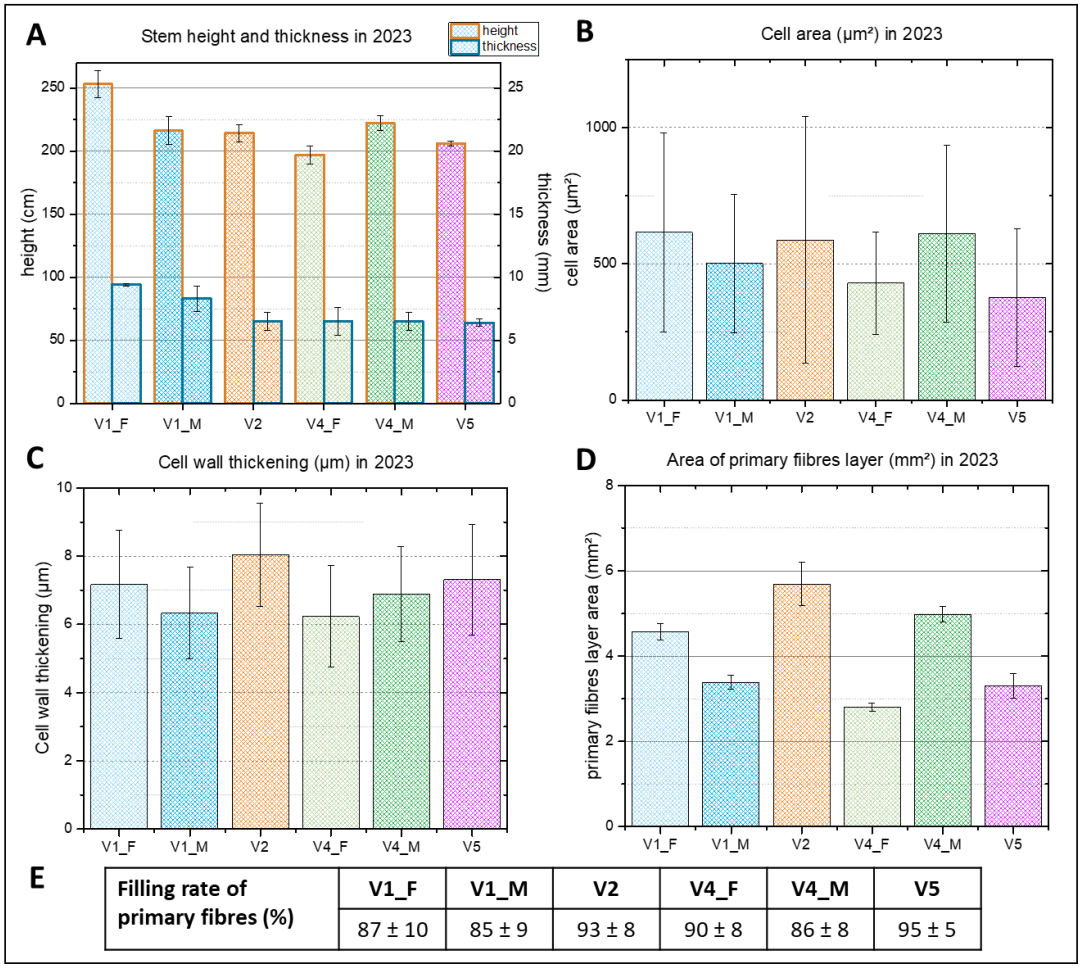
E

Filling rate of primary fibres (%)	V1_F	V1_M	V2	V3_F	V3_M	V4_I
	81 ± 10	89 ± 6	86 ± 7	78 ± 10	86 ± 8	91 ± 6

Figure 2.32: Properties of stems and fibres in 2021 (04/08/22 – 85 days after sowing) and 2022 (03/08/22 – 82 days after sowing). A: height and thickness of the tested stems; B: area of the fibres cross-section; C: thickness of the fibre cell wall; D: area of the primary-fibre layer within the stem cross-section, E: filling rate of primary fibres.

Thus, by comparing the studied varieties, the V4 has thin fibres with an advanced stage of cell wall thickening and a large proportion of primary fibres layer and thus seems the most appropriate for fibre applications. If we consider the global fibres surface, including primary and secondary fibres, the late variety V4 got a proportion of fibres twice as high as the variety V1.

In 2023, observations of the stem cross-sections could be done on samples from August, the 2nd (76 days after sowing). Results in terms of filling rate of primary fibres, stem height and thickness, cell area and primary fibres layer area are reported in Figure 2.33. In 2023, a high self-thinning was observed in variety V1, resulting in more dead plants or very short plants of about 1 m-height. Therefore, the other stems grew higher and bigger as observed in Figure 2.33.A. In 2023, the variety V5 was grown and the variety V3 was not grown. The variety late-flowering monoecious V5 reveals the highest filling rate, reaching 95% and the lowest cell area compared to the other varieties. It reveals promising results for textile long fibres applications. It is interesting to note that the two monoecious varieties V2 and V5 reveal similar size of stems and similar filling rates but opposite characteristics in terms of cell area value and primary fibres layer.



E

Filling rate of primary fibres (%)	V1_F	V1_M	V2	V4_F	V4_M	V5
	87 ± 10	85 ± 9	93 ± 8	90 ± 8	86 ± 8	95 ± 5

Figure 2.33: Properties of stems and fibres in 2023 (02/08/23 – 76 days after sowing). A: height and thickness of the tested stems; B: area of the fibres cross-section; C: thickness of the fibre cell wall; D: area of the primary-fibre layer within the stem cross-section, E: filling rate of primary fibres.

2.3.3. Extracted Fibres

Another important aspect for farmers when selecting a textile hemp variety is the quantity and the quality of the extracted long fibres after scutching.

2.3.3.1. Yields

It is important to notice that the higher the filling rate of the fibres, the heavier the fibres, and it would give higher long fibre yields to the farmer especially if the area of primary fibres is high. The stem yield of the year 2021 and the bast fibres yield after industrial scutching are gathered in Table 2.6.

Table 2.6: Harvesting and scutching yields in 2021.

Variety	Stem yield 2021 (Mg/ha)	Long fibres yield 2021 (kg/ha)	Short fibres yield 2021 (kg/ha)	Total fibres yield 2021 (kg/ha)
V1	6.6	538	2,360	3,058
V2	8.6	969	2,162	3,172
V3	8.4	957	2,170	3,135
V4	8.2	1,253	2,166	3,541

We observed that V4 has the highest long fibres yield, reaching 1,253 kg/ha, corresponding to a long fibre content of 16.8%. The lowest long fibres yields were obtained for V1 and V2. A large gap in flowering time between the male plants and the female plants of V1 created heterogeneity in plant characteristics. The male plants were taller and dry on the day of harvest while female plants were at full flowering, creating cutting problems during harvesting. Likewise, the retting process was not as homogenous as for the other varieties, and it has affected the fibres yields. Among all the varieties, the variety V4 produced the highest total fibres yield, reaching 3,541 kg/ha.

In 2022, the stem yields were lower than in 2021. The harvesting and scutching yields are presented in Table 2.7. The highest stem yield was found for the latest-flowering variety V4, reaching 6.4 Mg/ha. In 2022, the long fibres yield was the highest for the variety V2, reaching 1,030 kg/ha, while the total fibres yield was the highest for the variety V4, reaching 2,433 kg/ha. The variety V3 engendered difficulties during processing due to too long and large stems and over retting, causing a lot of waste and low fibre yields.

Table 2.7: Harvesting and scutching yields in 2022.

Variety	Stem yield 2022 (Mg/ha)	Long fibres yield 2022 (kg/ha)	Short fibres yield 2022 (kg/ha)	Total fibres yield 2022 (kg/ha)
V1	5.8	853	1,360	2,213
V2	6.1	1,030	1,260	2,332
V3	5.9	113	2,000	2,113
V4	6.4	608	1,680	2,433
V5	5.5	763	1,316	2,142

2.3.3.2. Fineness

The fineness of the extracted fibres after scutching and after hackling was characterised by FibreShape software as described in Section 2.2.3 of this chapter.

The width distribution of fibres bundles of batches from 2021 and 2022 are presented in Figure 2.34 and Figure 2.35. It is interesting to notice that the rankings according to the median or the global distribution differs from the scutched fibres to the hackled fibres. In 2021, a higher refinement of the bottom part of the stems is observed compared to the top part. The refinement depends on the division potential of the fibres and large fibre bundles might be easily separable in the bottom parts, or else the fibre largest bundles might be lost during hackling process.

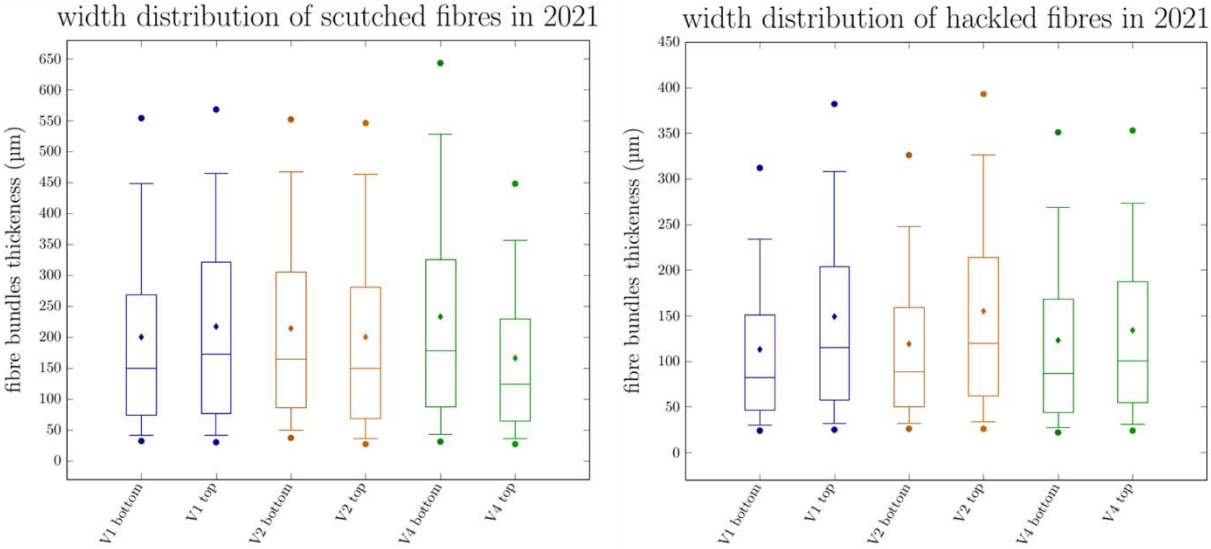


Figure 2.34: Width distribution before (left) and after (right) hackling in 2021 batches.

In 2022, a larger panel of varieties was compared. The dioecious (V3) and the monoecious (V5) varieties were also analysed.

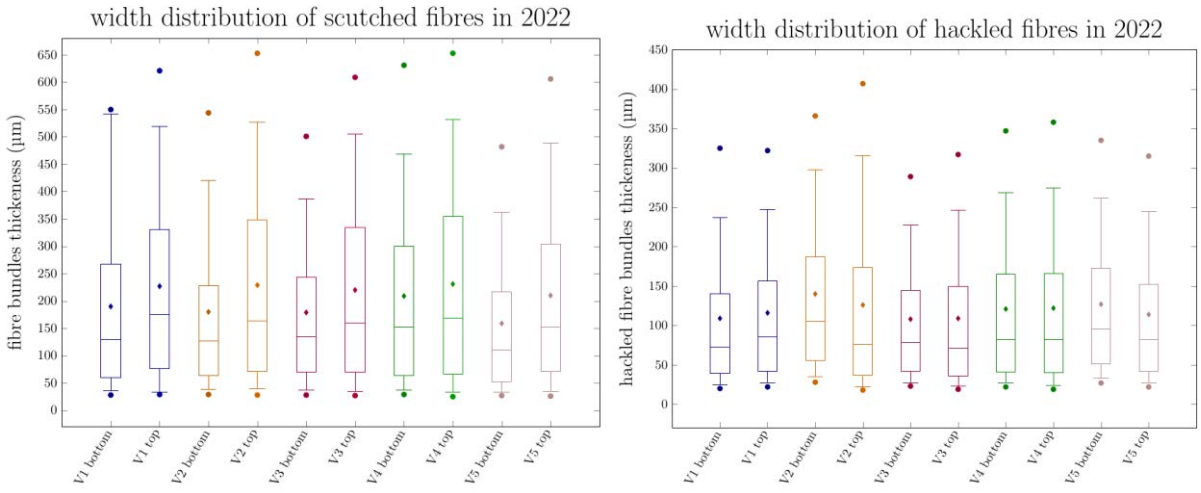


Figure 2.35: Width distribution before and after hackling in 2022 batches.

In 2022, a higher refinement was found in the upper part. After hackling the differences in width distribution between lower and upper parts are finally reduced. The variety V2 possesses a fibre bundle thickness distribution characteristic of thicker fibres compared to the other varieties and it is coherent with the fact that the size of the fibre cell area was also the biggest (Figure 2.32).

2.3.3.3. Organo-leptic

The scutched long fibres were evaluated by four experts in textiles. They use organo-leptic measurement to evaluate the quality of the fibres (Figure 2.36). The organo-leptic measurements are based on several criteria:

- Nature: density, brightness
- Colour: retting assessment
- Strength: by successive pulling
- Fineness: division of fibres
- Homogeneity: cleanliness



Figure 2.36: Organo-leptic evaluation, from LCBio.

Batch homogeneity is the most important criterion for scutchers, combers and spinners. According to the results, they can be estimated to a type of yarn by scutcher or comber: Type 1: < 12 Nm, Type 2: [12; 15] Nm, Type 3: [15; 18] Nm, Type 4: [18; 20] Nm, Type 3^{+}: [18; 20] Nm, Type 4: 24 Nm, Type 4^{+}: 26 Nm, Type 5: [26; 28] Nm, Type 5^{+}: [28; 30] Nm, Type 6: > 30 Nm. Yarns ranging from type 3 to type 5 gather 60% of yarn market share. The Table 2.8 presents the results from 2021 and 2022 batches. The judgements are detailed by criteria and the global evaluation is estimated as a type. These results are only preliminary judgements and can vary from those of spinners. The bottom parts are globally more appreciated than the top parts. The variety V2 was the most appreciated.

Table 2.8: Results of organo-leptic evaluation of 2022 batches.

	2022						2021
	Nature	Colour	Strength	Fineness	Homogeneity	Type	Type
V1 bottom	4	5	4	3	4	4	3
V1 top	3.5	5	3	3	3	3+	3
V2 bottom	4	5	3	3	4	5	5
V2 top	3	5	4	3	4	3	4
V3 bottom	3	5	3	3	2	3	4
V3 top	4	5	2	4	4	4	2
V4 bottom	3	5	3	3	4	3	3
V4 top	3	5	3	3	4	3	3
V5 bottom	4	5	3	4	4	4	-
V5 top	3	5	3	3	4	3	-

2.4. Conclusions

For textile applications, high quality hemp fibres and high fibre yield are sought. The time of harvesting and the choice of variety are crucial point to obtain textile grade fibres. The present research work highlights that harvesting a late-flowering variety before flowering time is advantageous in the latitude of Plain de Caen for obtaining high-quality fibres. Late flowering hemp has a longer vegetative phase than earlier flowering varieties and are usually recommended to obtain high yield of biomass and occasionally for high yield of long fibres. In this work, late-flowering varieties are advised to obtain high yields and high-quality fibres if dew retting is well performed. Harvesting before the plants flowering (end of July, early August) is suggested so that dew retting can be operate with a sufficient time before the arrival of too rainy days and humid air from early September. The late-flowering varieties in this study get secondary cell wall with a filling rate higher than 75% before the plants start flowering and often are higher than 90%. The size of its fibres are rather small (good for fine yarn manufacturing) and it has a large primary fibre layer cross-section suggesting a great potential for high fibre yield. Finally, after extraction through a scutching system, a late-flowering variety reached a high long fibre yield, reaching 1.25 Mg/ha. Finally, the selection of cultivars intended for textile fibre purpose in the territory of Normandy is not limited to early flowering varieties, but rather emphasises late flowering cultivars. Harvesting hemp plants prior to flowering should not be an impediment to varietal selection.

On another side, it was said that dioecious varieties would be more adapted for textile applications than monoecious varieties because in the past female and male were generally harvested separately and male plants were used for the finest fabrics while female plants were used for the coarse fabrics. In fact, generally, the male plants were pulled up earlier than the female plants that were harvested at seed maturity for dual applications. Male plants were harvested when the pollen shed. In this study, two types of experiments were carried out to observe the fibre properties from male and female plants: the analysis of fibre properties from male and female fibres extracted separately and the study of the stem cross-sections during the plant growth. These findings provide additional support to the results found in the literature. Male plants generally produced higher fibre content than female plants. The size of the fibre bundles was not strictly finer for male plants on the contrary to what could be expected from the historic literature. Female plants show more secondary fibres than males, and higher mechanical properties at seed maturity. The filling rate of the primary fibres is higher for male or for female depending on the year but this order is common between the varieties. On the first and second year, males from all varieties are higher filled than the female and the opposite happened on the third year. Moreover, the comparison between various dioecious and monoecious varieties revealed notable differences in terms of fibres bundles fineness after scutching or hackling. One monoecious variety was found to possess higher fibre bundle thickness distribution and larger elementary fibres than dioecious varieties, while the other monoecious has the smallest ones. Compared to dioecious varieties, the monoecious varieties are reported in literature to be more uniform in plant height and stem production. In this study, considering the size and the growth stage of the plants, the monoecious variety displays more homogenous results, as some dioecious varieties could have a large shift between male and female flowering.

Even if the production of a stable monoecious variety is challenging for seed breeder as the monoecious plant sometimes tends to return to its natural state of dioecy, the monoecious varieties has been rather developed by breeders in the past decade. The dioecious varieties were less investigated and consequently less seeds are available for large seed parcels. Since only female plants produce seeds, dioecious varieties are economically less profitable than monoecious varieties for hemp breeders and the prices of the seeds are

extremely higher. As the predominantly monoecious varieties were developed by breeders and were available in the seed catalogue, a monoecious variety will be rather used in the following Chapter to discuss about the industrial feasibility of hemp cultivation in Normandy.

Chapter 3. Development of a Reliable Industrial Long Hemp Fibres Extraction

This second chapter focuses on the transformation of the long hemp fibres. First of all, the objectives of a new crop implementation in Normandy will be detailed. The development of new harvesting equipment and an adapted fibres extraction line are targeted. The extraction is based on a flax scutching line. The process parameters such as the cutting height, the material moisture content and the scutching speeds are studied. Results in terms of feasibility and efficiency of the process are presented. Characterisations of the final products are carried out and finally an economic approach is performed.

3.1. Objectives of the Development of a New Textile Chain in France and Europe

A strong demand for natural fibres for industrial textile applications is emerging. The increasing interest in low environmental impact and local products suggests a high potential for reliable local textile fibre production in Europe. The two current main sources of textiles, cotton and synthetic fibres, are associated with major environmental issues. In contrast to synthetic fibres, which consume petroleum and are responsible for the dissemination of microplastics in the environment, and cotton fibres, which require large amounts of water, pesticides, and fertilisers, the cultivation and use of hemp and flax fibres have a more favourable environmental balance *(Ellen MacArthur Foundation, 2017; European Environment Agency, 2022; van der Werf and Turunen, 2008)*. In areas where textile flax is cultivated, such as Normandy, the limit of the surface cultivated has been reached. Textile flax is grown on fields with a good nutriments rate where retting is possible and in the crop rotation, flax is grown every seven years to limit the development of diseases. In addition, a new crop to substitute the sugar beetroot, which encounters disease problems in Normandy, is sought. Hemp was identified as a good opportunity. Since the beginning of the 21st century, hemp has experienced renewed interest. Its environmentally friendly aspect makes it particularly attractive and an interesting alternative to the main sources of textile fibres. Indeed, the hemp crop requires little input and provides a positive impact on crop rotation, in addition to revealing phytoremediation potential. The production of hemp in Europe increased by 70% from 2013 to 2018, barely growing 54,481 hectares in 2020, including 36% in France *(Interchanvre, 2022a).* Nowadays, hemp is grown primarily for its seeds, its short or randomly oriented fibres, and its shives, the woody core of the straw. These three markets, which include hemp feed, paper, insulation, mulching, litter applications, and the construction industry, are well established in France *(EIHA, 2018)*. In addition, a strong demand for hemp fibres for industrial textile applications is emerging as a complement to flax fibres which are in ever-increasing demand. There is a significant opportunity for reliable local textile fibre production in Europe, given the growing desire in locally produced, low-impact goods. Studies on small-scale trials show that hemp fibres have great potential for textile applications and clothing manufacturing *(Amaducci et al., 2008b; Vandepitte et al., 2020; Westerhuis, 2016).*

As a consequence of the limited production in the second half of the twentieth century, hemp cultivation and particularly its harvesting have not been modernised, and no complete mechanised harvest chain for long textile fibres is nowadays industrially available. Adapted devices, therefore, need to be developed. One apparent solution to establishing large-scale long fibres hemp production would be to rely on the flax model from the field to fibre extraction using scutching and hackling processes. A temperate zone with mild and humid summers is highly favourable for this technique. This type of climate is typical of Normandy, where dew-retting has been widely used for flax for many years. Normandy produced 527,900 Mg of textile flax in 2019 and made France the world leader in the production of textile flax *(Chambre d'Agriculture Normandie, 2020).* In this region, farmers have acquired strong knowledge of dew-retting. In Normandy, flax is harvested in mid-July, whereas hemp for textile purposes is harvested later. At the time of flowering when the fibre yield is supposed to be high, the primary

fibres reach an advanced stage of maturity, and the presence of secondary fibres is expected to be low (Chapter 2).

Rainy weather usually occurs at the end of the summer in Normandy from early September, and this may cause issues regarding the baling of dew-retted stems as these need to be stored dry. It is therefore safe to bale and store before the beginning of September, before the level of humidity in the air and on the ground turns out to be too high and the soil potentially too muddy. Straw bales that are rolled up in wet conditions could engender the development of post-retting within the bale. To deal with this request, it is necessary to harvest the hemp as early as possible so that there is sufficient time for the dew-retting process to take place and for the straws to dry enough before being baled. For the test, an early-flowering hemp variety was chosen to target a harvesting at full flowering.

This work is included in a project willing to progressively demonstrate the feasibility of operating a complete industrial value chain using as much as possible the flax machine elements already present in the Normandy territory for the generation of long hemp textile grade fibres. The extraction of long hemp fibres in the manner of the flax model is investigated. A large-scale field trial was performed in conjunction with industrial scutching investigations. Long fibres and scutching by-product yields, fibre strength and stiffness and fibres bundles fineness of hemp are evaluated as well as the economic interest of such a production which would come as a complement of flax fibres which are in constant increasing demand *(European flax, 2021; Statistiques FAO, 2022).*

3.2. Materials and Implementation of a New Industrial Scutching Line

During scutching, the harvested one-meter-long flax stems pass through a succession of breaking rollers and a beating system that crush the stems and remove the shives to extract the long fibres respectively. In order to adapt the existing flax scutching line to hemp, some settings of the processing line need to be adapted, and hemp stems, which can grow up to more than two-metres high, need to arrive in one-metre length. Then two parts of the stems are separately processed.

In 2021, at the beginning of this thesis, only a few machines designed for hemp are able to obtain two separate windrows from the top and bottom parts of the hemp plant. For an efficient hemp harvest, the machines require effective cutting elements that can cut hemp stems at desired heights (combining or not with a pulling up element), a conveyor system that can send the hemp stems out in an adequate time and a mechanism that can drop off the stems in parallel and aligned manner. Mechanised harvesting trials were carried out in 2020 and before, using specific mowers developed in China or Italy (BCS Group, Abbiategrasso, Italy). This type of machine holds a single sickle bar, then a cutting disc machine is used in succession to separate the stems into two portions of one metre. These machines are presented in Chapter 1, Section 1.2.2. However, they are still not fully adapted to large-scale, efficient harvesting, as these machines are more designed for small-scale local productions. For large-scale industrial production, a prototype (Hyler Sativa 200A) capable of harvesting 2 $ha.h^{-1}$ was developed in 2021 by the Hyler company (Hyler, Sint-Baafs-Vijve, Belgium) (Figure 3.1). It is working with two cutting blades with adjustable height, a ventilated system that maintains the cut stem in rows, a conveyor system that throws the bottom part of the stem on one side and the top part on the other side. The top flowers are cut with a third blade. Finally, a conveyor belt lays two parallel rows of aligned stems, one for the bottom and one for the top, down on the field. This prototype, purchased by “Coopérative Linière du Nord de Caen”

and supported by the Normandy region, was tested with global success at the end of the 2021 summer and has been used for large-scale harvesting in summer 2022. In summer 2023, seven Hyler machines were used in Western Europe.



Figure 3.1: Hyler Sativa hemp harvesting machine.

Another harvesting machine has been developed in 2022 by Cretes company (Valtech Group, Wevelgem, Belgium) (Figure 3.2). It is a device that fastens at the tractor's rear. The tractor rolls over the rows just made and the blades are placed offset from the tractor. Two blades cut the stems in two portions. The upper stem parts are cut and placed on the lower stem parts, thereby rows with a mix of top and bottom parts are produced by this technique. The flower tops are separated from the upper stem parts by passing through another blade and put on the ground between the stem rows.



Figure 3.2: Hemp harvesting machine from Cretes company.

During retting, the stems are turned over with a flax turning machine to homogenise the retting process among the rows. The windrows are baled dried, with the same machine used for flax and then stored in a closed barn (Figure 3.3).

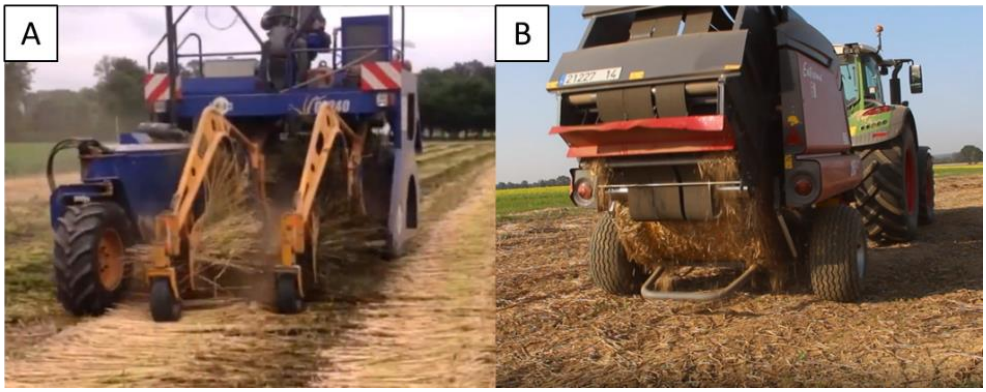


Figure 3.3: A returning system and baling system used for flax and hemp.

3.2.1. Conditions in Normandy

From sowing to harvesting dates, the weather was cool with an average minimum and maximum temperature of 12 °C and 22 °C, a relative humidity between 59% and 87% and a total precipitation of 79.5 mm which was distributed throughout the period (78 days) as illustrated in Figure 3.4. The growing conditions were therefore very good if one compares to some studies which indicate that optimum stem yields typically require 50 cm to 70 cm of available water especially during vegetative growth in the Northern hemisphere. These studies also mention that hemp grows best when the mean daily air temperatures are between 16 °C and 27 °C *(Adesina et al., 2020; Fike, 2016).*

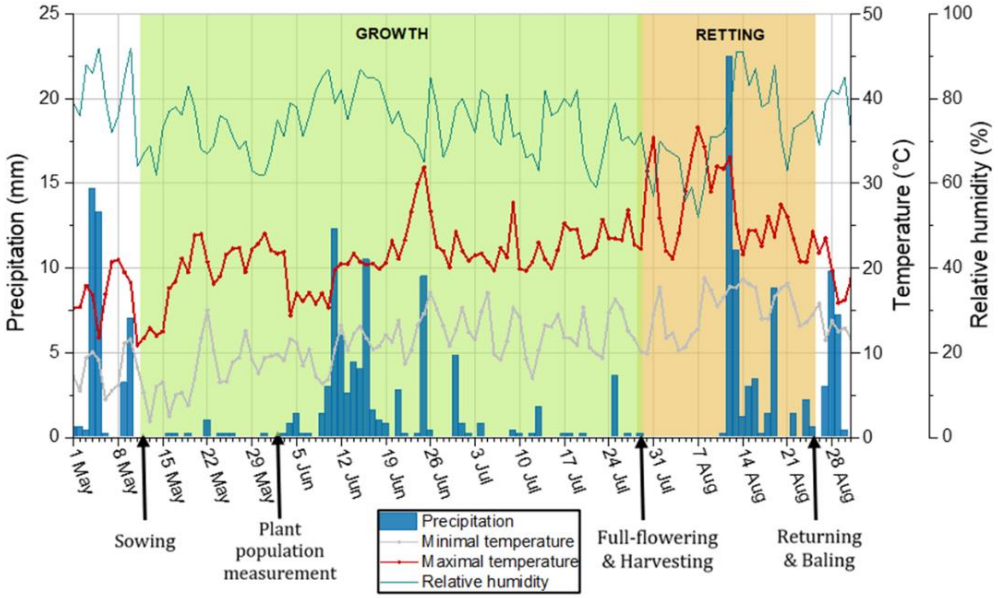


Figure 3.4: Weather data in the field trial location in 2020 during the growth of the hemp plants and field retting: daily precipitation, relative humidity and average daily temperature.

Following the flax model, stems may be dew-retted before being scutched. The stems are laid down in parallel windrows in the field after harvesting, and the biodegradation of pectic cements, facilitating the separation of fibres from the rest of the plant, is achieved by microorganisms in the soil. This technique has a low environmental and economic impact but is highly dependent on climatic conditions. The control of this process is therefore difficult, and it may have a high influence on fibre quality. Excessive retting weakens the fibres, whereas under-retting does not favour sufficient degradation of the binder between fibres. In this case, coarse fibre bundles, not suitable for fine textile manufacturing, are produced.

At the beginning of retting, the weather was dry and mild with a total precipitation of 0.4 mm. After thirteen days high precipitation of 33.5 mm occurred within two days and thereafter an alternance of humid and dry weather with cool temperature gave ideal conditions for the field retting *(Bleuze et al., 2020; Liu et al., 2015a)*.

The monoecious variety V2, mentioned in Chapter 2 and named USO-31, was chosen in this study. It is an early-flowering variety which was already tested with success in preliminary tests on small batches in Normandy. The variety is registered in the European Union seed catalogue and is widely available. At this latitude, the full flowering occurs between the last week of July and the first week of August. The field trial was carried out in 2020 at the farm of Henri Pomikal, located in Martragny on the Plain of Caen, Normandy, France (49° 15’ 4.739 N – 0° 35’ 48.80” O). The plot was set up as part of the textile hemp project conducted by “Lin et Chanvre Bio” cluster and supported by the Agence de l’Eau de Seine-Normandie. “Lin et Chanvre Bio” provided the technical itinerary, handled the purchase of seeds and performed the monitoring of the crops. The seeds were provided by Hemp-it company (Hemp-it, France) and were sown with a density of 500 units per m² (80 kg/ha) with an average growing capacity estimated at 60% according to the past years observations. A high-sowing density was chosen to obtain high plants density with expected thin straws to favour high bast primary fibre content and low amounts of secondary fibres *(Amaducci et al., 2002a; Struik et al., 2000)* as demonstrated in Chapter 2.

Sowing was carried out on 12 May 2020 with an “Amazone” seed drill combined with a “Cultimix” rotary harrow. The sowing depth was 2 cm and the distance of the inter-row was

12 cm. The variety was sown on a large-scale plot of 15,000 m². Prior to sowing; two stale seed beds were carried out on 15 March and 15 April 2020 in order to prevent self-propagating plant growth and favour the warm-up of the soil. The preparation of the soil is an essential process in hemp cultivation to grow quality hemp plants. The soil type of the field trial is loamy which is considered as a favourable texture for hemp growth *(Adesina et al., 2020).* Before sowing, the nitrogen residue was evaluated to be 97 kg N/ha on the top of the 0.60 m soil layer and no nitrogen has been added subsequently. The plant population at the early growth stage, measured on 2 June 2020, was 275 plants.m^{-2}.

Harvesting was carried out on 29 July 2020 at full flowering using a single hemp sickle bar mower bought by the "Lin et Chanvre Bio" cluster (illustrated in Chapter 1) on the Chinese market. At that time, new harvesting machines such as Hyler machines or Cretes machines were not developed yet. Stems were cut about 10 cm above the soil and laid down parallel for field-retting. The retting degree was checked regularly by expert assessment (H. Pomikal) on the basis of organoleptic criteria and scutching assays of a small mass of stems. Thus, the colour, the homogeneity, the resistance and the level of separation of the bast fibres were observed through a hand-tensile test and the quality of the fibre extraction was controlled during scutching preliminary tests. Stems were field-retted 27 days, which is consistent with other studies presenting a high microbial spreading and good fibre properties *(Bleuze et al., 2020; Mazian et al., 2019; Réquilé et al., 2021).*

The average plant height measured at harvest time was (180 ± 10) cm. The stem diameter was evaluated at 20 cm above ground level, on 50 stems and the average thickness was (5.2 ± 1.1) mm. The thickness distribution is presented on Figure 3.5.

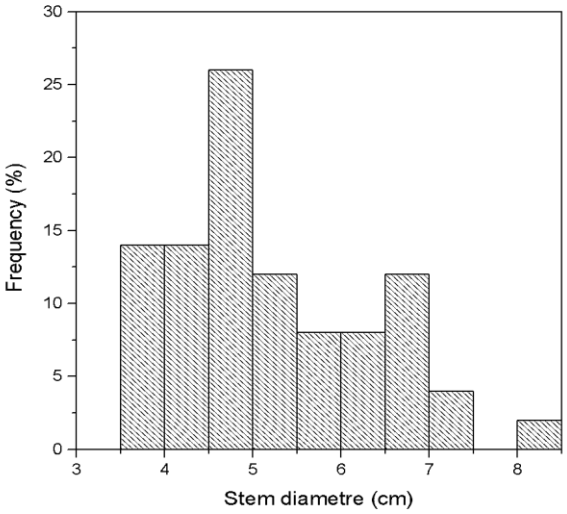


Figure 3.5: Distribution of stem diameter.

3.2.2. Yields

3.2.2.1. Stem Yield

The top and bottom parts of the hemp stems may be treated separately in the future as the fibre properties and homogeneity are expected to be different. The morphology of the fibres, as well as the mechanical properties and cell wall composition, differ from the bottom to the top parts. According to Liu et al., a significantly higher lignin content was observed in the bottom part of the stem compared to the top part at seed maturity, and the strength and

stiffness of fibre bundles were higher for samples from the top and middle parts *(Liu et al., 2015a).* According to *(Amaducci et al., 2005)* and *(Müssig and Amaducci, 2018),* the fibre diameter and maturity tend to decrease from the lower part to the upper part of the stem. Additionally, the bottom is more affected by the presence of secondary fibres compared to the top. The latter are bast fibres that are smaller and more lignified than primary fibres and are unwanted for textile applications *(Abot, 2010)*. As mentioned in chapter 1, their presence was reported to hamper the production of fine and homogeneous yarns during spinning. Secondary fibre formation occurs in late plant development at the bottom of the stem and accelerates around flowering, depending on stem height and diameter. The height of the secondary fibre front is directly correlated with the plant size. It depends on the varieties and is most likely related to the need to increase the resistance of the stem as the weight of the plant increases *(Hernandez et al., 2007b; Westerhuis et al., 2019).*

The stem and fibre yields of this study are presented in Figure 3.6.

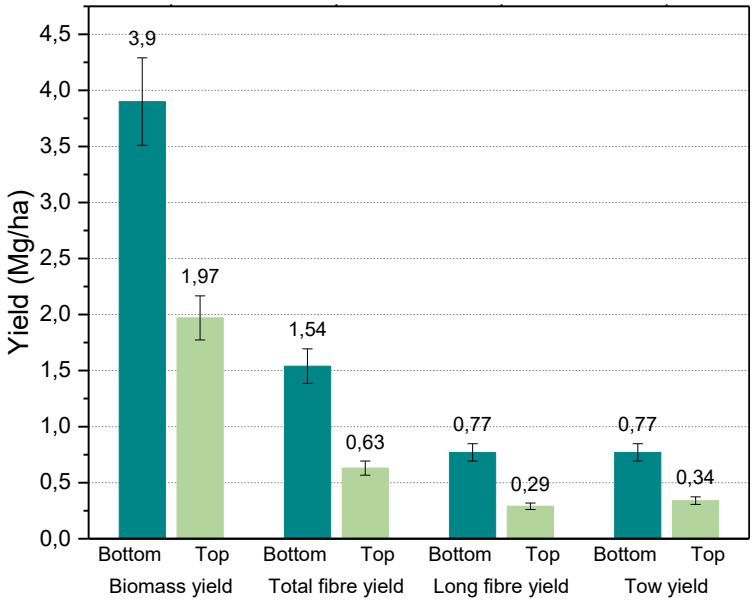


Figure 3.6: Stem and fibre yields following industrial harvesting and scutching. Statistical Student test was carried between bottom and top parts: letter a indicates no significant difference between the two parts; letter b indicates a significant difference between the two parts.

Stem yield evaluated by measuring the total weight of bales of dew-retted dry stems was equal to 6.71 t/ha. The present stem yield value is almost twice as high as the estimated value (3.63 Mg/ha) for the same variety cultivated in Flanders region (Belgium) and harvested at flowering time *(Vandepitte et al., 2020).* It is comparable to the yield of the other varieties they studied (mean equal to 5.68 Mg/ha). It is interesting to note that the field trial of this study was performed in a large plot (15,000 m²) and this should provide a more accurate estimation of the yield and reduces the field edge effects as the stem density was globally well homogeneous. The effects of plant density on USO-31 stem morphology are in agreement with previous research reporting that plants grown at lower density tend to have thicker stems *(Westerhuis, 2016).* In Vandepitte et al. (2020) studies, for a targeted density of 240 plants/m^2, an average stem diameter of 6.5 mm was obtained, whereas for a targeted density of 275 plants/m^2 in the present study, the measured diameter is 5.2 mm *(Vandepitte et al., 2020)*. The stem height was identical in both studies.

Other works conducted on the variety USO-31 have been reported in the literature with different growing conditions. Sankari (2000) found a stem yield of 4.70 Mg/ha in Finland and

Adamovics et al. (2016) obtained a stem yield of 13.59 Mg/ha in Latvia *(Adamovics et al., 2016; Sankari, 2000).* In both cases, hemp was grown at higher latitude than Normandy and was harvested when matured seeds appeared. Besides a lower stem yield, Sankari (2000) reported a lower stem length (132 cm) and a lower diameter (4.7 mm) whereas Adamovics et al. (2016) reported higher stem lengths, with the average among varieties varying between 258 cm and 318 cm depending on nitrogen supply (Adamovics et al., 2016). Both studies evidenced a clear positive correlation between plant height and stem yield. High and large stems are, however, not advised for textile applications since they favour the presence of secondary fibres which are unwanted for producing fine yarns due to their lower length and itching problem that may arise. Hernandez et al. (2007) and Westerhuis et al. (2019) reported that the secondary fibres reach only up to the second or the third internode as long as the plant is still below 225 cm height and 1 cm in stem diameter or as long as the plant has not started flowering *(Hernandez et al., 2007b; Westerhuis et al., 2019)*. The height of the secondary fibres front is getting higher with the increase of the weight of the plants. Moreover, the decrease of plant density, therefore the increase of stem thickness, induces an increase of the thickness of the secondary-fibre layer *(Amaducci et al., 2008b).*

The stem yields of the bottom part, the top part and a mixed part of top and bottom parts were respectively 3.90 Mg/ha, 1.97 Mg/ha and 0.84 Mg/ha. The mixed part corresponds to the edge of the field trial where the top and the bottom could not be mechanically separated. The stem yield from the bottom portion was twice as high as from the top part. This tendency was also observed by Amaducci et al. on other varieties *(Amaducci et al., 2008b)*.

3.2.2.2. Long Fibre Yield

The total amount of stems from the field trial was scutched on the flax industrial line. Total fibre yield was 37.2% including a long fibres yield of 17.4% (1.17 Mg/ha) and scutching tows yield of 19.8% (1.33 Mg/ha). These results were obtained by adjusting the settings of the flax scutching line for hemp extraction such as reducing the conveyor speed during crushing and the rotation speed during beating.The results are consistent with previous studies on hemp fibre yields with an industrial scutching line *(Grégoire et al., 2021)*. Musio et al. (2018) used an industrial flax scutching line with flax parameters for extracting dew-retted stems and found a long fibre yield ranging from 2.1% to 15.6% depending on varieties, while Vandepitte et al. (2020) found long fibre yields of 17.3% in average depending on varieties by using a flax scutching line with a reduction in turbine speed *(Musio et al., 2018; Vandepitte et al., 2020).* These results highlight the correlation between the increase in fibre yields and the reduction in the speed of the flax industrial chain.

The fibres extraction from the top and the bottom part were studied separately. The long fibre yield of the top part was 0.289 Mg/ha and the bottom part was 0.769 Mg/ha. The bottom part gathered 73% of the long fibre production whereas the top part had only 27%. On the previous part, it is indicated that the stem mass of the bottom part is twice as high as one of the top part (Figure 3.6). This is, of course, due to the fact that the diameter of the top part decreases as a function of the height of the plant. The respective total fibre yield is also higher in the bottom part than in the top part of the stems, 39.5% and 32.1% respectively. At that stage, a ton of bottom part stems provides more fibres than the top part and should therefore be valued at a different price. Moreover, the long fibre yield is higher for the bottom part than in the top part, 19.7% and 14.7% respectively. So, for an equivalent mass of stem coming from the bottom part, higher quantities of fibres are expected to be extracted with a higher percentage of long fibres. By using chemical extraction, the same distribution of long fibres between the top and bottom portions was reported by Amaducci et al. (2008) among a large range of varieties *(Amaducci et al., 2008b).* Through manual decortication, Mediavilla et al.

(2001) stated that approximate 54% of the fibres were accumulated in the bottom part, 34% in the middle, and only 12% in the top part of the stem *(Mediavilla et al., 2001).*

3.2.1. Fibre Quality

3.2.1.1. Organo-leptic Analysis

To introduce hemp fibres into the fashion textile sector, fibres should be spinnable into fine yarns. The majority of the demand on the market targets yarns between 24 Nm and 26 Nm. Nm is the metric number, which is the yarn length in metres per gram. The higher the Nm, the finer the yarns. The finer the yarns that can be spun, the higher the value of the material. The fibres were evaluated by experts on the basis of organoleptic tests typically used for controlling flax quality. Criteria such as the nature of fibres (density, flexibility, brightness), the homogeneity, the colour, the strength, and the fineness are assessed to estimate the potential of separation (divisibility) and transformation of the fibres. Batch homogeneity is the most important criterion for combers and spinners. According to first observations, the level of retting has an important impact on the divisibility and compared to flax, the colour range of the hemp fibres seems narrower and colour seems to have little effect on the quality. According to a spinner evaluation, the fibres obtained after scutching are suitable to form yarns in the targeted range of Nm 24 and Nm 26 that is suitable for a very large range of garment fabrics.

3.2.1.2. Fineness

For the production of high-value fibres for textile applications, fibre thickness is an important trait when the objective is the manufacturing of fine yarns. To refine and parallelise the fibres, scutched fibre samples were hackled. On average, the hackling yield was close to 50%. This value is comparable to the study of Musio et al. (2018) using an industrial discontinuous hackling system *(Musio et al., 2018).* They found a hackling yield of fibres of (48.5 ± 2.0)% and showed that hackled fibre bundles were finer than scutched ones. In the present studies, the thickness was measured using the method described in Section 2.4 on hackled fibres to have a global representation of the fibre population. Figure 3.7 provides a statistical descriptive approach by representing a box plot with the percentile points, quartile points, median, and arithmetic mean of the width distribution.

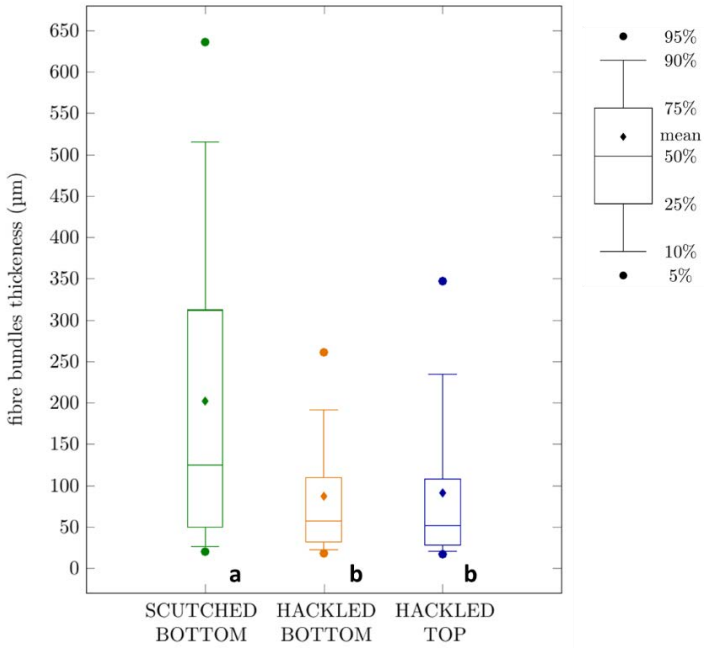


Figure 3.7: Width distribution of scutched fibre bundles and hackled fibre bundles from the bottom and top parts of the stems. Statistical Student test, described in Annex 3: letter a indicates no significant difference, and letter b indicates a significant difference.

The fibre bundles from the top part present a wider distribution of thickness compared to the bottom part. A larger amount of coarse fibre bundles is obtained on the top part since 10% of the thickest fibres are over 235 µm for the top part and 191 µm for the bottom part. However, a larger amount of well-separated fibres or thin fibre bundles is obtained in the top part than in the bottom part since 50% of the fibre population in the top part reaches a diameter lower than 51 µm whereas it is 57 µm for the bottom part.

During spinning preparation, the largest fibre bundles will be separated since a doubling drawing system reduces the width of fibre bundles and removes the thickest ones. Müssig and Amaducci (2018) also compared the fibre bundle thickness at different heights in the stem with the Fibreshape system and found that after chemical extraction and mechanical refinement, fewer coarser fibre bundles are obtained in the top part than the bottom part *(Müssig and Amaducci, 2018).*

3.2.1.3. Mechanical Properties

Tensile properties obtained from stress–strain curves is another relevant parameter to consider when evaluating high-quality long fibres. Fibre strength is required in the spinning process to prevent fibre breakage. The tensile properties of individual fibres extracted from the top and bottom parts of stems are presented in Figure 3.8. The stress–strain curves of flax or hemp fibres may exhibit different tensile behaviours, either linear or non-linear. The non-linear behaviour, typical of natural fibres, has been described in many studies and is attributed to the microfibrils realignments *(Duval et al., 2011; Placet et al., 2014a).* However, the stage of the stress–strain curve at which this phenomenon occurs is not well defined.

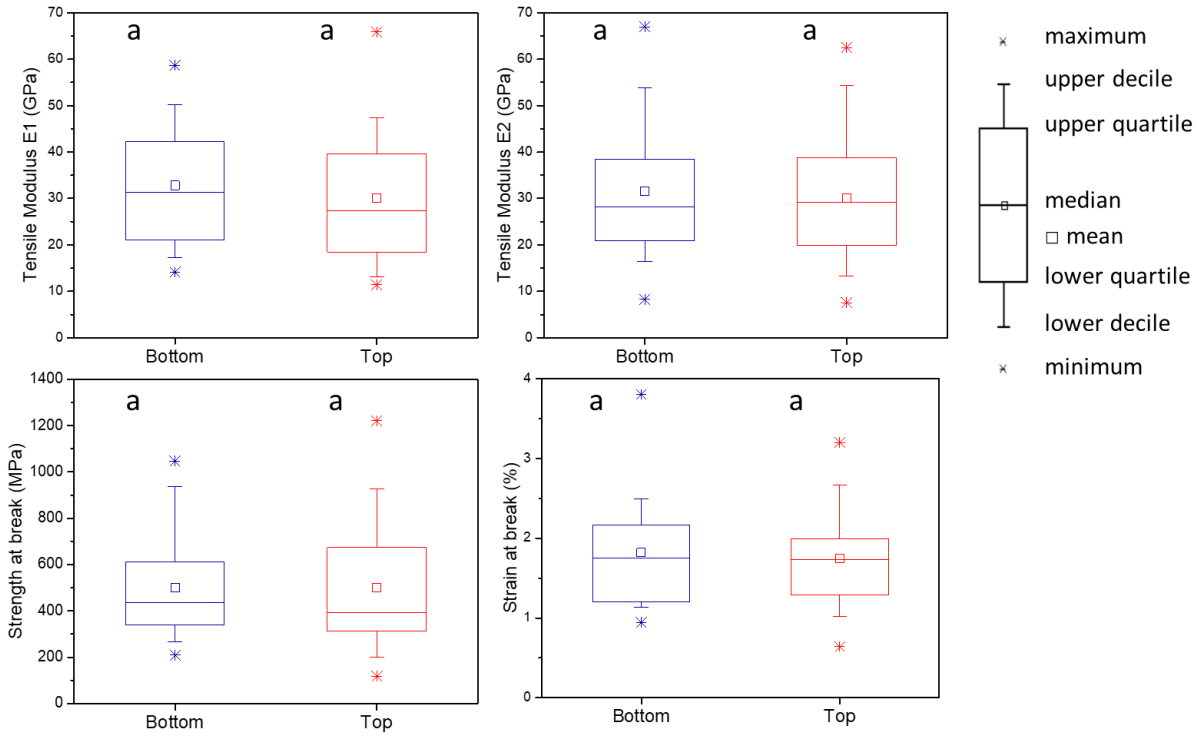


Figure 3.8: Tensile properties of single fibres. Bottom and top parts. Statistical Student test, described in Annex 3: letter a indicates no significant difference between the bottom and top parts.

No significant differences are observed between the tensile properties of fibres extracted from the top and bottom parts. The strains at breaking point are equal to (1.8 ± 0.7)% and (1.7 ± 0.6)%, respectively, for the bottom and top portions, and the strengths at breaking point are similar to a higher dispersion for the top part (bottom: (501 ± 243) MPa, top: (500 ± 286)MPa). The tensile modulus values are lower for the top part than for the bottom part, with tensile modulus E1 and E2 mean values both equal to (30.0 ± 14.4) GPa for the top on average and (32.2 ± 13.5) GPa for the bottom portion.

The comparison of tensile property values with those from the literature must be performed carefully, as the characterised object (elementary fibres or fibre bundles) may be different. The methods, the conditions, and the treatment may also have an important effect on the results. Table 3.1.A gather the tensile values of the variety USO-31 hemp fibres, while Table 3.1.B shows the tensile values of different hemp varieties analysed only on single fibres. The ultimate tensile strength in this study reaches lower values than the ones collected from the literature, and the tensile modulus values are also lower than expected. However, those values are suitable for textile applications and sufficient for spinning processing.

Table 3.1: Tensile properties of hemp fibres. A: with USO-31 variety; B: on single fibres only.

	Authors	Hemp Variety	Testing Conditions	Tensile Modulus E (GPa)	Strength at Break (MPa)	Strain at Break (%)
A	Vandepitte et al., 2020	USO-31	fibres bundle	-	597	-
	Liu et al., 2015	USO-31	fibres bundle	34.9 ±4.4	954 ± 162	6.2 ±1.2
	Sankari, 2000	USO-31	fibres bundle	-	860 ± 670	4.3 ±4.4
B	Marrot et al., 2013	Fedora 17	single fibre	35.5 ±17.3	889 ± 472	2.6 ±2.2
	Placet et al., 2012	-	single fibre	24.7 ±11.4	636 ± 253	2.1 ±0.7
	Grégoire et al., 2021	Futura 75	single fibre	49 ± 19	875 ± 367	-
	This study	USO 31	single fibre	32.2 ±13.5	500 ± 286	1.7 ±0.6

The values found in the present study can be a consequence of the industrial extraction process. During industrial scutching, more damage may be generated on the fibres, and a decrease in tensile properties may be observed *(Placet et al., 2017; Richely et al., 2021).* Grégoire et al. (2021) found that the tensile properties of hemp fibres extracted using an industrial scutching line with traditional flax set-ups were 35% lower than the ones measured on fibres extracted using lab-scale equipment with softer process parameters *(Grégoire et al., 2021).* The less aggressive the decortication step, the better the mechanical properties of the fibres *(Müssig, 2020).* One may therefore wonder if the relatively lower tensile properties mentioned in this work compared to the ones reported in the literature and mainly obtained on manually extracted fibres are due to a too severe extraction environment, even though values found by Grégoire et al. (2021) with more aggressive industrial settings were higher than the ones of the study.

3.2.1.4. Cross-sections Analysis

Another characteristic, the lack of maturity of the fibres, may also play an important role in explaining the low tensile properties. An analysis of the fibre's maturity was carried out. Maturation of the fibres proceeds from bottom to top and from the outer to the inner part of the stem with a cell wall deposition and post-synthetic modification, and its evolution varies with

agronomic practices *(Amaducci et al., 2008b; T. E. Chernova et al., 2018; Mediavilla et al., 2001).* Hemp-matured fibres are characterised by thick cell walls and a small lumen. Placet et al. (2012) showed that the modulus is dependent on the size of the lumen *(Placet et al., 2012a).* For a constant fibre diameter, an increase in the lumen size results in a decrease in the modulus values.

The cross-section of the stem in the bottom part was analysed to get a visual notion of the cell wall thickness. The images shown in Figure 3.9 also illustrate that secondary fibres are not present in the stem. The lumen of the fibres is highly visible and represents a large proportion of the fibre area. This means that the fibres, even if harvested at full flowering, are not fully filled and are presumably not representative of matured fibres. In chapter 2, it was reported that some variety with early or intermediate flowering time, did not reach their fully filled state at full-flowering, as it could be supposed. It seems to be a competition between the energy supply for flowering and seed production and the energy supply for the vegetative and fibres development. The variety V2 (USO-31) was filled at 75% on the 3rd of August 2021 while later-flowering varieties were higher filled. In 2020, the harvest happened on the 28th of July, then it can be supposed that the fibres did not reach their full-filled state. The low thickness of the cell walls probably explains the modest tensile properties of the single fibres tested in this work.

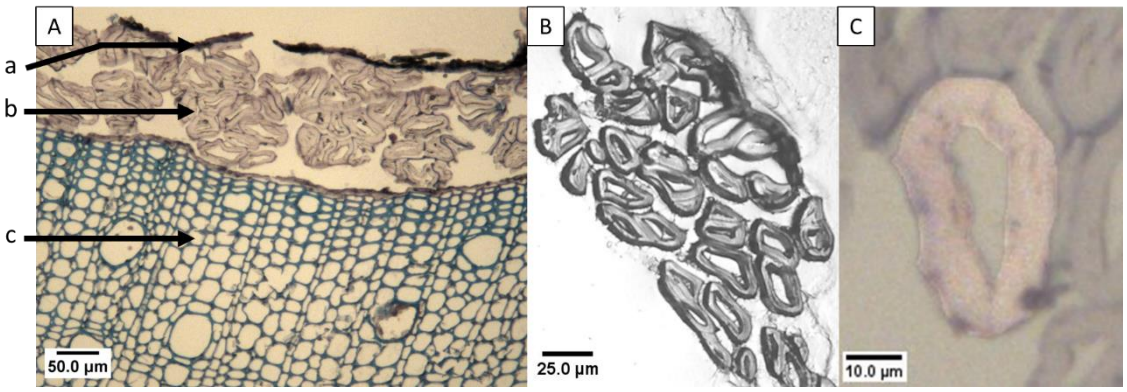


Figure 3.9: Images of a cross-section of a peripheral part of a stem, including a: epidermis, b: bast fibres, c: xylem (A), fibre bundles (B), and a single fibre (C).

3.3. Progressive Improvement over Several Years

The extraction process was monitored for three successive years. A fourth year is still in progress at the present time. The cultivated area of the variety USO-31 was gradually enlarged each year, starting from 1.5 ha in 2020 to 10 ha in 2023. During the period between sowing and harvesting of the four years, the cumulative precipitation was 80 mm, 166 mm, 118 mm, and 149 mm respectively. The Table 3.2 gathered the obtained yields over the studied years.

Table 3.2: surface cultivated and yields from 2020 to 2023 for USO-31 variety.

Year	Surface (ha)	Stem yield (Mg/ha)	Long fibre yield (kg/ha)
2020	1.5 ha	6.71	1,167
2021	2.3 ha	8.61	969
2022	5.8 ha	6.12	1,071
2023	10 ha	/	/

The 2021 harvest reveals a higher stem yield and a lower long fibres yield compared to the previous and following year. These differences were mainly due to the pulling up of the stems instead of a cut and changes in scutching parameters. In 2021, the roots were also baled and scutched with the stems, producing a higher weight of biomass but creating difficulties at the extraction. Moreover, numerous tests were done over the three years to optimise the production rate and adjust the industrial scutching speed parameters. According to Grégoire et al. working at lab scale, lower speed parameters lead to higher scutching yield without decreasing the extracted fibres quality *(Grégoire, 2020; Grégoire et al., 2021)*. Their studies revealed that an increase in the long fibre yield from 9.2% to 28.9% was possible at the lab scale after scutching by adjusting the feed and beating speeds to three times lower than those traditionally used in the industrial scutching module. Moreover, very large quantities of long fibres were observed to fall in tows during industrial extraction using traditional settings. In 2020, the reduction in speed has been adapted at an industrial scale. The transport chain speed and the first turbine speed were reduced by 25% and then fixed to 80 m/min and 170 rpm, respectively. The decrease in speeds was limited compared to that was studied by Grégoire et al. (2021) and could be even more reduced to improve the long fibre yield and preserve the quality of the fibres. However, the production rate of the line must be considered and cannot be reduced too much, as otherwise the cost of scutching would rise too much.

In order to improve the production rate and limit the tows to long fibres ratio, new set of tests were done in 2021 and 2022 (Table 3.3). It was postulated that the high rate of scutching tow fibres might be due to a too aggressive beating of the system that breaks the long fibres into tow fibres. Then a turbine speed was reduced to limit the aggressivity of the blades on the fibres. As a result, the long fibres yield increased by decreasing the turbine speed. The speed limits were investigated. It has been reported that at too low speed, fibres rolled up round the turbine axes, some bottom parts of the stem were not well extracted and also the engine could not cool and consequently overheated. Outbreaks of fire could be caused. It was not possible to set the turbines speed below 125 rpm. Consequently, to limit the beating time, it is suggested to increase the transport chain speed. It is also suggested to set lower transport chain speed for the bottom than for the top part, as the separation of the shives and the extraction of fibres from the top part was easier than the bottom part, especially when roots are present. Over the three years, several points for an efficiency scutching were brought out: (1) a homogeneous and dense stem bed distribution is necessary. A regular amount of stem per metres without any blanks within the stem bed is advised since there are more losses of fibres at the onset and the end of the stem bed; (2) bales need to be well tight to ease the rolling out and the formation of the stem beds. When the rolling out of the bales is not done correctly by the machine, the stems are not properly placed on the belt and the technicians have to replace them which is an exhausting work; (3) The roots need to be removed from the stems as their presence leads to low quality extracted fibres; (4) The length of the stems needs to be in adequation to the scutching machine size. It was seen that too long stems create disordered and too short stems causes losses; (5) A too high beating speed causes break of fibres (6) A too slow beating speed causes an inefficient division of the fibres, coiling of the fibres around the axis and risk of fire outbreak.

Table 3.3: Overview of the stem and scutching yields from 2020 to 2022.

Year	Stem yield (kg/ha)	Long fibres yield (kg/ha)	Long fibres content (%)	Tows yield (kg/ha)	Tows content (%)	Total fibres yield (kg/ha)	Total fibres content (%)	Production rate (kg/h)
2020	6,707	1,027	17.4	1,329	19.8	2,356	37.2	1,180
2021	8,563	1,010	11.8	2,162	25.3	3,172	37.0	1,700

2022	6,122	1,071	17.5	1,261	20.6	2,332	38.1	1,500

In this section, the scutching speed settings and the stems bed arrangement were investigated on industrial line and have been seen to influence the final yields of fibres, the extracted fibres quality and the output. In the next section, parameters linked to the state of the raw material are studied at lab scale. The moisture of the stems and the extraction of the furthest bottom part are considered.

3.3.1. Humidity Parameters

In Normandy, the relative humidity varies between 40% and 99% with an average around 80%. The relative humidity has an impact on the moisture content of the plants. It was said that it is better not to scutch on a rainy day or humid day and hemp scutching should be carried out after winter when the moisture decreases. In this study, analysis of moisture contents at a given RH were carried out on the stem, the fibre individually and on the woody part individually. The tests were done by Dynamic Vapour Sorption (DVS) in water solvent at an isotherm of 24.8 °C. They were conducted over a relative humidity range from 0% to 90%, at 15% intervals and with a sample quantity of about 5 mg. The sorption and desorption of the stem and the fibres are presented in Figure 3.10.

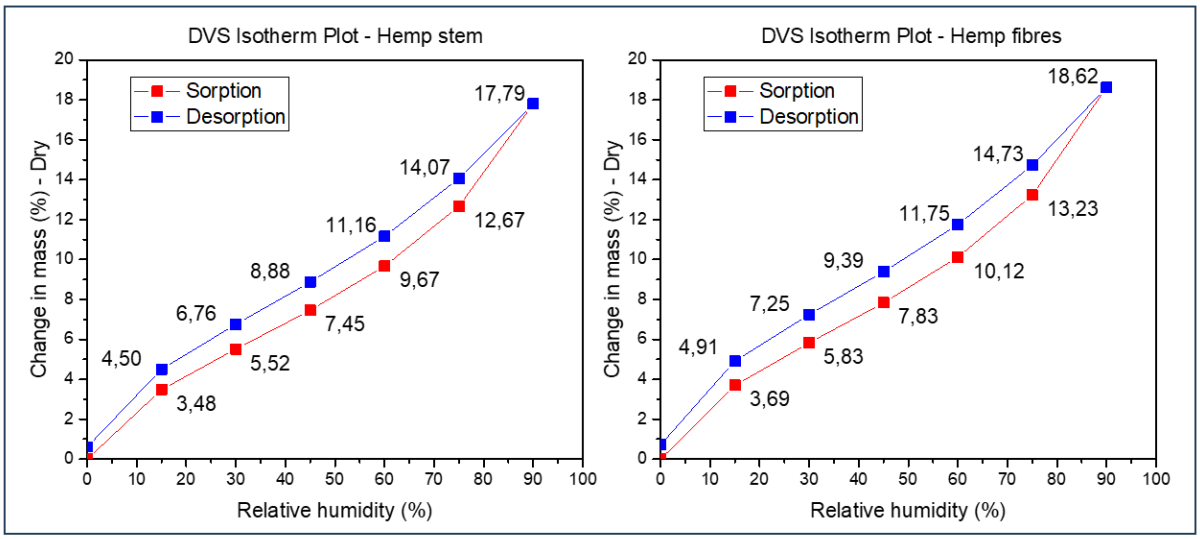


Figure 3.10: Dynamic Vapour Sorption isotherm plot for hemp stem and hemp fibres.

It is interesting to note that stem and fibre moisture contents depend on the relative humidity of the ambient atmosphere and the sign of the moisture change. Two hemp samples of the same type will possess different moisture contents at the same relative humidity depending on its previous conditioning. This is a characteristic phenomenon of hemp and lignocellulosic fibres called moisture hysteresis. When the fibre is moved from wet to dry conditions, desorption takes place and the fibre moisture content is higher in comparison to the value obtained with the same air conditions with sorption phenomenon, when the fibre changes from dry to wet conditions *(Zimniewska, 2022)*. The origins of this phenomenon are still debated in literature. Several authors attribute it to softening and polymer arrangement processes related to water interactions with amorphous biopolymers present in the cell walls *(Baltazar-y-Jimenez and Bismarck, 2007; Salmén and Larsson, 2018)*.

Fibre moisture content affects fibre properties. It has an impact on fibre behaviour during tensile deformation (changes on apparent fibre stiffness, strength, elongation, type of

rupture) *(Placet et al., 2012a)*. It causes swelling and can generate stress and local damages when fibres are embedded into a matrix *(Garat et al., 2020)*. It also has an effect on processing efficiency. According to Zimniewska et al., the optimal value of fibre moisture content for hackling and scutching would be between 10% and 12% *(Zimniewska, 2022).*

In this work, lab-scutching tests were performed with two varieties: Santhica and male Carmagnola. About 500 g of stems of 80 cm long was placed at controlled humidity and temperature for at least three weeks until the stem weight remains steady.

Three different values of relative humidity were chosen: 45% RH, 75% RH and 90% RH according to the relative humidity occurring throughout the year (Table 3.4). All the scutching tests were carried out at the ambient atmosphere, 22 °C and 50% RH. The stems were directly scutched after being taken out of the controlled room and a sample was collected to measure the stems moisture.

Table 3.4: Stem moisture content effect on scutching fibre yield.

Conditioning relative humidity (%)	Mean stem moisture content (%)	Mean long fibre yield (%)
45	8.9	21.9 ± 4.5
75	12.0	22.6 ± 0.1
90	13.5	18.4 ± 1.6

In this work, the efficiency of the scutching process was studied for stem moisture contents of 8.9%, 12% and 13.5%. The highest yield in terms of long fibres was found with stems containing 12% of moisture content, corresponding to a controlled ambient atmosphere of 75% RH. However, no significant differences were found between all the trials. Trial with fibres conditioned at 60% RH, corresponding to a stem moisture content from 10% to 10.5%, could not be performed. During the industrial scutching test, done in January or February in Coopérative Linière du Nord de Caen, the relative humidity was about 80%. It would be advised to wait for less humid days to optimise the fibre extraction efficiency.

3.3.2. Adjusting the Cutting Height: Study of Roots

New machines are developed to make the harvesting of hemp easier, and one concern is the effective length of the stems. The stems have to fit into the flax scutching machine and so each portion should measure between 80 cm to 100 cm long to obtain the best extraction efficiency. Long portions lead to higher yield and better incomes. In this way, the closer the stems are cut from the soil, the better it would be. Moreover, the tall stubble, the basal part of straws left in the field after harvest, could hamper the cut hemp plants from coming into contact with the soil during retting. Humidity and development of microorganisms can be impacted and retting modified. On another side, in the study of Westerhuis et al., the authors suggested taking off only the middle portion of the stem to obtain a higher homogeneity in fibre quality. They reported that the more mature the stem, the higher the lower blade should cut in order to avoid the presence of secondary fibres and then decrease the extracted fibres quality *(Westerhuis et al., 2019).* Generally, the stubble height is set around 15 cm to 20 cm above the soil. So, a portion of primary fibres is lost. The study of the fibres in the first centimetres of the plant height was carried out to adjust the height of the cut.

One trial of pull up was made and stems were scutched with the roots. As a result, the scutching process encountered difficulties due to the resistance of the roots and the inhomogeneity of the stem bed. The roots are thicker and harder than the stem portions. The extracted fibres revealed large parts of residues sticking to the fibres (Figure 3.11) and other

parts were broken due to roots resistance. In fact, in the past, the plants were pulled up and then deprived from the root, ligneous part which does not present valuable fibres (Barral, 1864).



Figure 3.11: Scutched fibres of hemp plants with roots.

A trial was conducted only on the 20 cm portion above the roots. It is interesting to notice that many fibres are still present in this part and could be valorised. In the above 10 cm, the fibres could get separated while in the lower 10 cm they contain much residue as observed on Figure 3.11.

In order to lose as little as possible valuable fibres, it would be interesting to cut the stems very low or pull up the stems and cut them a few centimetres above the root part. Cross-sections were carried out in the lower portion of the stems in order to estimate the quality of the fibres at 5 cm above the roots.

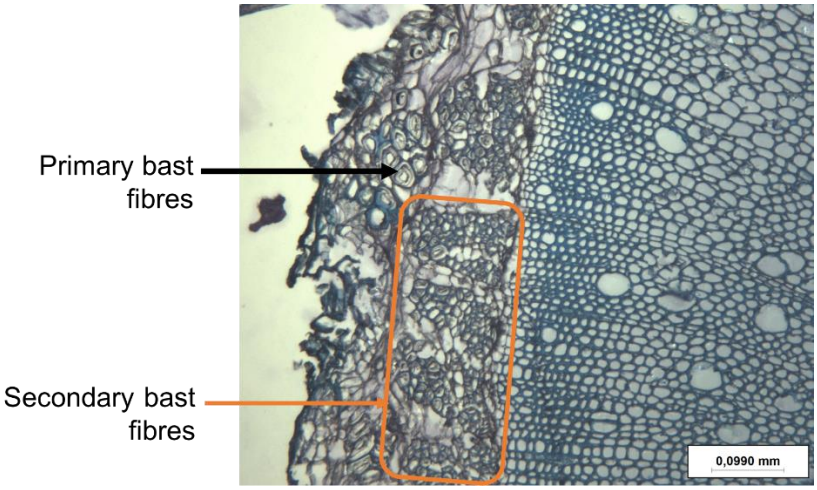


Figure 3.12: Cross-section of hemp stem taken at 5 cm above the root.

In Figure 3.12, the cross-section of a 5 cm height cut is presented and shows that the majority of the bast fibres are secondary fibres. More than 70% of bast fibres area corresponds to secondary fibres in this cross-section.

According to this study, the roots and the first centimetres of the stems (until the first 10 centimetres) should not be submitted to fibre extraction using the scutching process.

3.4. Economic Analysis

To complete the study of the industrial development of long hemp fibres in Normandy, an economic approach is relevant to determine if this cultivation is economically interesting and could provide sufficient income to farmers. The costs of cultivation and fibre extraction have been investigated. The prices chosen for this case study result from estimations based on textile flax market. It is, of course, worth mentioning that prices can change over the years. On the same model as flax, hemp farmers would be paid according to the quantity and quality of long fibres, tows, and shives. The higher the quality, the higher the price. In this study, the price of the long fibres is estimated to be 3 €/kg based on the 2020s Chinese market prices, independently from the quality of the fibres. Further investigations, considering the charges and the valorisation of the co-products, provide a better illustration of the economic approach of long fibre production. It is summarised in Figure 3.13.

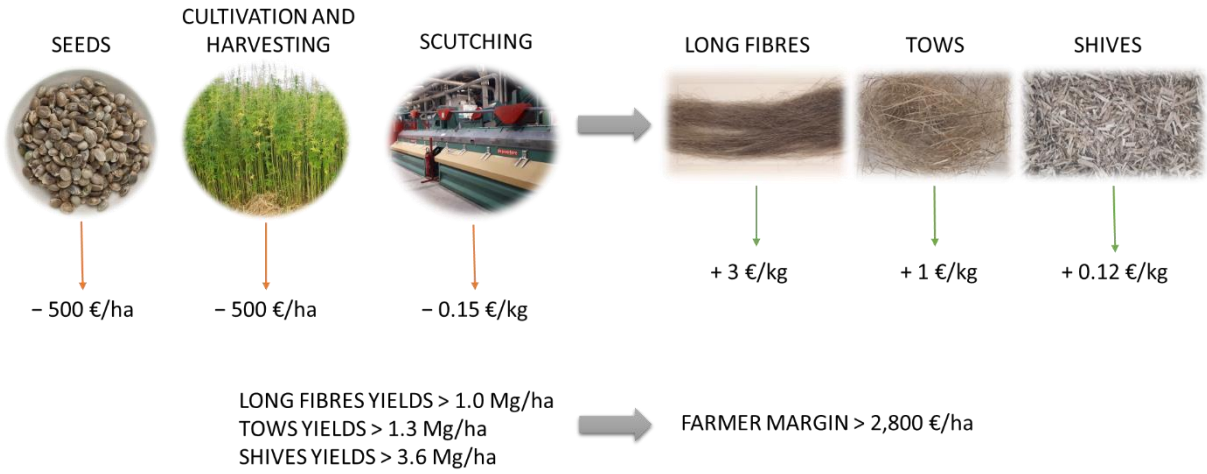


Figure 3.13: Schematic view of the production costs and farmer income in the case of 2020 harvest and market price.

The cost of the seeds is evaluated at 500 €/ha for this variety, the cultivation and harvesting costs at 500 €/ha, and the scutching process at 150 €/tons of stems. The profits from co-products, tows and shives, are estimated to 1 €/kg and 0.12 €/kg respectively. In 2020, the average long fibre yield was 1.17 Mg/ha from which about 0.14 Mg/ha were of low quality and were estimated to have a lower price equal to the tow price. By adding products and production costs, the global income (gross income per hectare) is estimated to 2,980 €/ha.

In three years, market prices changed a lot. The inflation, the increase in energy prices (electricity, fuel), geopolitical and, climate issues have driven the global prices up. The selling price of long fibres is rising as well, due to a sharp increase in the demand from purchasers in China and India. A new estimation according to the average yield over the three years and the market prices in 2023 is proposed. The results are presented in Figure 3.14.

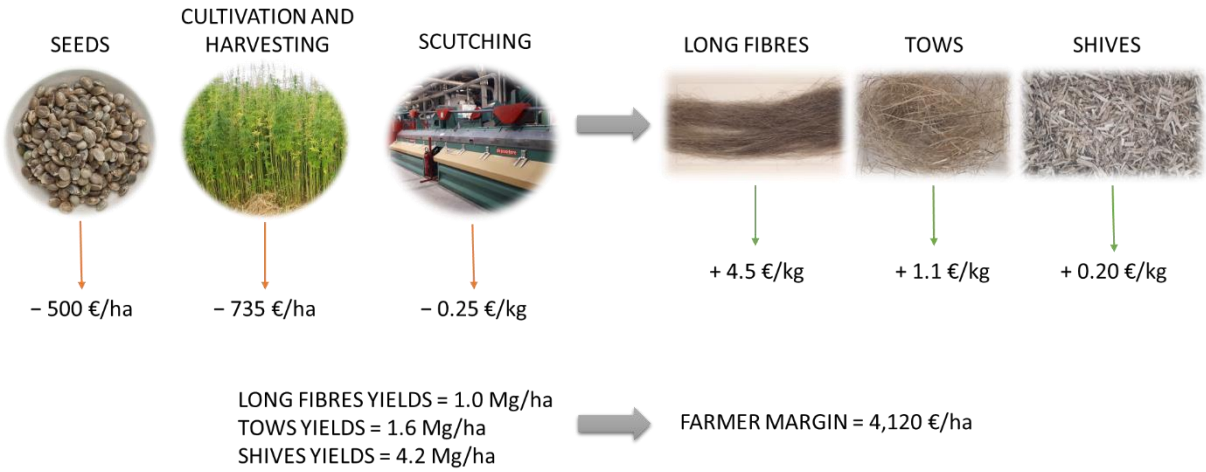


Figure 3.14: Schematic view of the production costs and farmer income in the case of 2023 market price and the average yields from 2020, 2021 and 2022.

These estimations were done for the cultivation of a monoecious variety. For now, most of dioecious varieties that are available on the seed catalogue are intended for seeds and flowers cultivation and are sold in low quantities. Hence, dioecious varieties are much more expensive due to seed availability. For example, seed price of monoecious variety is about 500 €/ha with high density seeding rates whereas seeds price of dioecious variety is at the moment more about 3,600 €/ha, considering 40 €/kg and a seeding rate of 90 kg/ha. If dioecious varieties are considered for long fibres harvesting, their prices will have to be close to the monoecious ones. An estimation of the economical approach is calculated in Figure 3.15, for the dioecious variety V4 studied in Chapter 2, with its highest long fibres yield, the 2023 market prices and the monoecious variety price. This estimation reveals great potential for this dioecious variety. All the estimations are calculated with a unique price for long fibres, however on fibres market the long fibres prices are matching with the quality of the fibres and can vary from more or less 1€/kg depending on fibres quality. According to chapter 2, even if the organo-leptic assessment attributes a low appreciation for the dioecious variety V4, V4 revealed a great potential for obtaining high quality fibres.

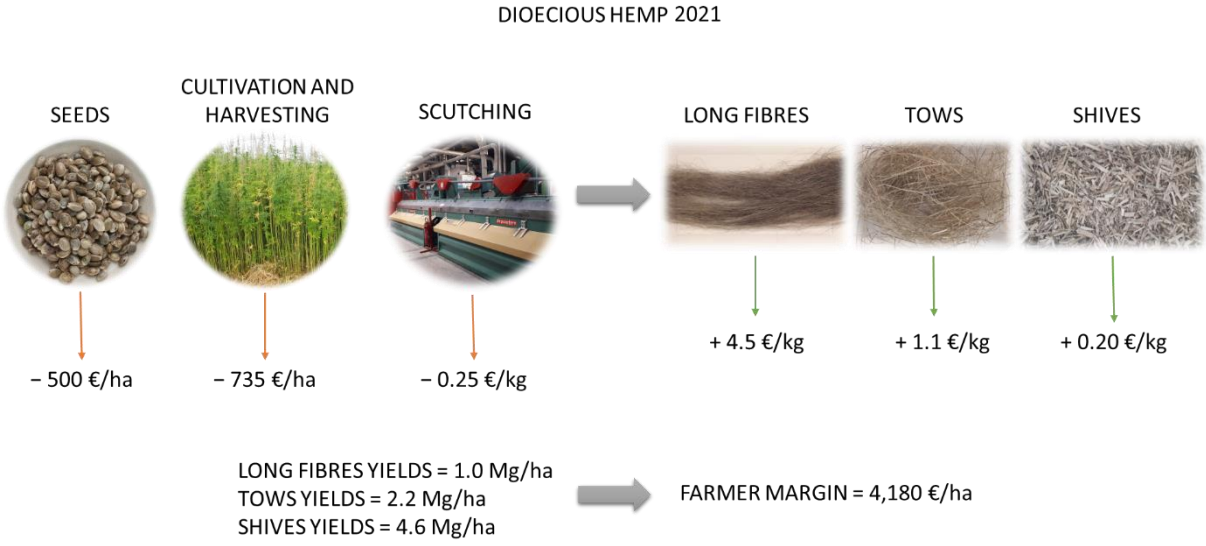


Figure 3.15: Schematic view of the production costs and farmer income in the case of 2023 market price and the yield of the V4 dioecious in 2021.

By comparison with other crops grown in Normandy (Table 3.5), hemp cultivation for fibres may be highly profitable in this area. However, further assessment should be performed over several years to complete a fully reliable study of the yields and economic aspects.

*Table 3.5: Gross income from main crops per hectare (ha), in Calvados, Normandy, from 2018 to 2022. *estimations. (CERFRANCE, 2023, 2021)*

Crop	2022 (€)*	2021 (€)	2020 (€)	2019 (€)	2018 (€)
Wheat	1289	1229	1011	1033	877
Rapeseed	1609	1477	835	782	713
Barley	1215	1073	698	659	667
Maize	1453	1343	886	680	781
Textile flax	-	1657	1335	2193	2315
Sugar beet	-	-	-	1334	1117
Potatoes	-	4666	4174	-	5153

3.5. Conclusions

The different elements proposed in this chapter demonstrate that the development of a textile dedicated hemp value chain, based on the flax machinery and know-how is on his way and is now industrial. The present work demonstrates that industrial volumes of hemp fibres can be processed from the field to textile grade scutched long fibres. Of course, some of the machine elements such as the hemp mowers or the disc cutters are still machines that need to be improved, but these can be considered for small local productions. For larger production, the industrial machine successfully used in 2022 and 2023 is adapted for full-scale industrial harvesting.

Beyond the global picture, the presented work showed that the hemp variety USO-31, can be grown on a large field trial, harvested and dew-retted with success. The experience of the dew-retting process combined with a very favourable climate is a clear advantage for Normandy farmers who can also use a large part of the flax dedicated equipment such as machines for returning and baling after the harvesting flax season. The dew-retted hemp straws were processed using flax scutching machines with adapted set-ups so that to obtain a good compromise between long fibre and production yields. Choosing adapted parameters such as the removal of the roots, the storage of the material before scutching in an atmosphere of 60% to 80% relative humidity, the good tightening of the round balls to obtain a homogeneous stem bed at the entrance of the scutching process and a low beating speed ensure a better output with quality fibres and high yields. More than 1.17 Mg/ha of long fibres were obtained in the frame of a crop that does not necessitate any phytosanitary treatment and in this case no fertiliser. The properties of fibres were found to be highly sufficient for the garment textile industry requirements and particularly for the European spinning industry that is in high demand from their client for fine hemp yarns.

The study also demonstrated the interest of growing hemp from an economic point of view as the cultivation of hemp by experienced flax farmers can lead to high range income in comparison to classical crops grown in the same area and this case study therefore shows good prospects for exponential expansion of hemp in Normandy.

Chapter 4.
Spinning of Fibres

This last chapter focuses on the spinning of fibres. Spinning consists in the production of yarns from raw materials. The fibres extracted from the hemp and oil-seed flax plants are used in this chapter as raw material for spinning. They are prepared and twisted to form a continuous yarn. In this chapter, first of all, the spinning of long hemp fibres is studied following the traditional flax wet spinning approach. The hemp obtained after the scutching extraction process described in the previous chapter is used, and the further steps are described in detail until the yarns are produced. The preparation steps consist mainly in hackling, drawing, roving, degumming and spinning. Yarns are spun at the laboratory scale and characterised in terms of evenness, tensile properties and linear mass. Industrial scale spinning is performed and complete the hemp transformation line from the field to the yarn.

Secondly, an investigation of the spinning of waste products is carried out. Flax agro-waste (oil-seed flax fibres) and hemp scutching tows are valorised for textile applications. The ability to produce yarns is studied at laboratory scale and characterisations of fibres and yarns in terms of chemical and morphological properties are performed. Finally, a semi-industrial scale is reached. The up-scaling process and the characterisations on the spun yarns and knitted fabrics are examined.

4.1. From the Long Fibres to the Yarns

When discussing about hemp criteria for textile applications, most of the research focuses on the growth of hemp, the extraction of the fibres and the resulting yields and the fibres quality without investigating the yarn properties and the quality of the following products. This study allows a complete monitoring from the plant growth to the yarn.

After their extraction, the long fibres undergo several steps before being transformed into yarns. Firstly, fibres are hackled with the objective to divide the fibre bundles into smaller entities, to align them and to remove the short fibres. The hackled fibres are assembled to form a continuous sliver that is drawn and homogenised before being twisted into a roving and winded. Generally, the rovings are submitted to a chemical treatment, called degumming to refine the fibres and remove some of the remaining components that stick the fibres bundles together. This step can be combined with bleaching to lighten the fibres colour and further divide the fibres. Finally, the treated materials are spun by wet spinning. These steps from the hackling to the yarn formation were studied at a laboratory scale.

The methodology carried out for long hemp fibres spinning at laboratory scale is illustrated in the flow-chart presented in Figure 4.1 and each step are detailed in the following sections.

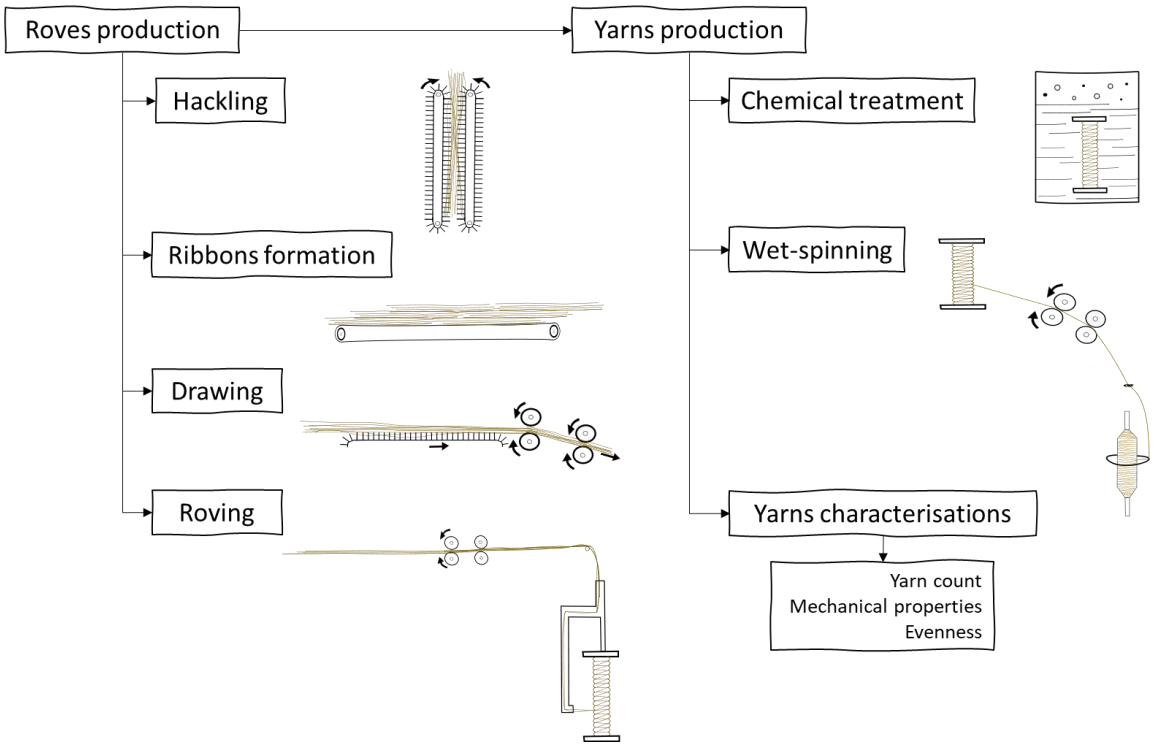


Figure 4.1: Flowchart of wet-spinning process of long hemp fibres at laboratory scale.

4.1.1. Roves Production

Hemp fibres were extracted at an industrial scutching line in “Coopérative Linière du Nord de Caen”. The extracted fibres were hackled on the Tap-root lab-device. The average hackling yield reached 50%. One kilogram of hackled fibres was obtained and were used to form a continuous sliver The sliver was made by laying the handful of hackled fibres on a preparation bench and overlapping handfuls of fibres with an overlap of 25 cm. A continuous and quite regular sliver of 40 g/m is obtained. The linear mass was chosen according to the linear mass of industrial textile flax samples. The preliminary step is very important to obtain a homogenous and regular yarns. Then the slivers were drawn, refined and homogenised on a drawing machine, called Mini-System manufactured by Linimpianti company (Linificio e Canapificio Nazionale, Bergamo, Italy). A belt of pins brings the fibres to a roller. The speed difference between the pin bed and the roller aligns, refines and stretches the slivers. The linear mass is divided by a factor 8. Six passages are done with a progressive refinement of the pins and a narrowing of the pins spacing and the rollers. At each step six slivers are assembled together to form the new initial slivers for the following step.

The evolution of the fibre bundles thickness was followed over the drawing process. An example of the evolution of a dioecious variety is presented in Figure 4.2. Fibres were extracted by separating the males from the female plants and the upper parts from the lower parts. The upper parts and the lower parts were mixed before drawing. A clear diminution of the average thickness is observed. The thickest bundles were stretched by the drawing system and separated into finer bundles.

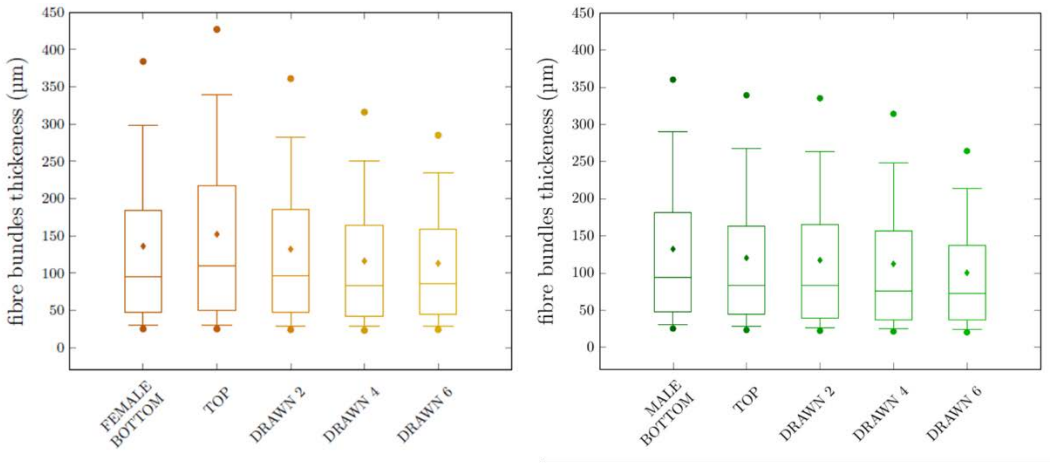


Figure 4.2: Width distribution of hemp female (A) and male (B) fibre bundles after hackling and drawing.

During the processes, the fibres can be damaged from coming into contact with metal needles and undergoing stress. Tensile tests were carried out before and after stretching to compare the tensile properties of the fibres.

Table 4.1: Tensile properties of elementary fibres from top and bottom part after hackling or drawing.

Variety	MALE bottom	MALE top	MALE drawn	FEMALE bottom	FEMALE top	FEMALE drawn
Strength (MPa)	624 ± 261	624 ± 250	622 ± 289	727 ± 343	687 ± 345	613 ± 332
Tensile Modulus (GPa)	29.7 ± 11.0	32.3 ± 11.7	29.7 ± 13.1	37.7 ± 14.1	32.6 ± 11.9	30.8 ± 14.8
Strain at break (%)	2.3 ± 1.0	2.0 ± 0.8	2.3 ± 1.1	2.0 ± 0.7	2.2 ± 0.6	2.1 ± 0.9

According to the tensile test results, the drawing frame step does not influence the tensile properties of male batches and a slight decrease in tensile properties is observed for the female batch.

At the final stage of the drawing, four slivers were assembled to form a final sliver of 4 g/m. This one was used to from a roving. A roving machine from Linimpianti company (Linificio e Canapificio Nazionale, Bergamo, Italy), is used to form rovings from the sliver. The sliver is drawn with a factor 10 and a light twist is applied to maintain the roving thread on a bobbin and to increase its tenacity. A roving thread of 0,40 g/m is obtained.

4.1.2. Yarns Production

4.1.2.1. Chemical Treatments

Following the mechanical preparation steps, a chemical treatment was performed. The goal of this process is to decrease the lignin and pectin content of the fibres. Both components are essential during the hemp growth but are not compatible for high-quality fibre processing, as they make the fibres stiffer, coarser, and hence more difficult to process. A dioecious variety (V4) was used to realise the tests.

Three different experiments were carried out:

- No treatment
- Alkaline treatment
- Alkaline and bleaching treatment

Alkaline treatments were carried out by immersing fibres into a solution of sodium hydroxide (NaOH) of 0.2 mol/L for 2 hours at 80 °C. Neutralisation is done with acetic acid solution. Bleaching is carried out by immersing the material in a hydrogen peroxide solution for 30 min at 60°C. To perform the treatments, roves were rolled up on an adapted 3D printed PETG plastic bobbins of 9 cm height containing holes in its centre to favour the liquid circulation. A batch ratio of 1/20 (w/V) was respected, and around 20 g of material were used.

The bleached product was lighter than the untreated and the alkaline treated materials. The mass losses of the material were around 15% after both treatments. However, these losses were also due to partial removal of the materials during manipulation and not only due to the chemical reaction. The thickness of the fibre bundles was measured after roving production and after treatments, and was compared with that of textile flax. The thickness distributions are presented in Figure 4.3.

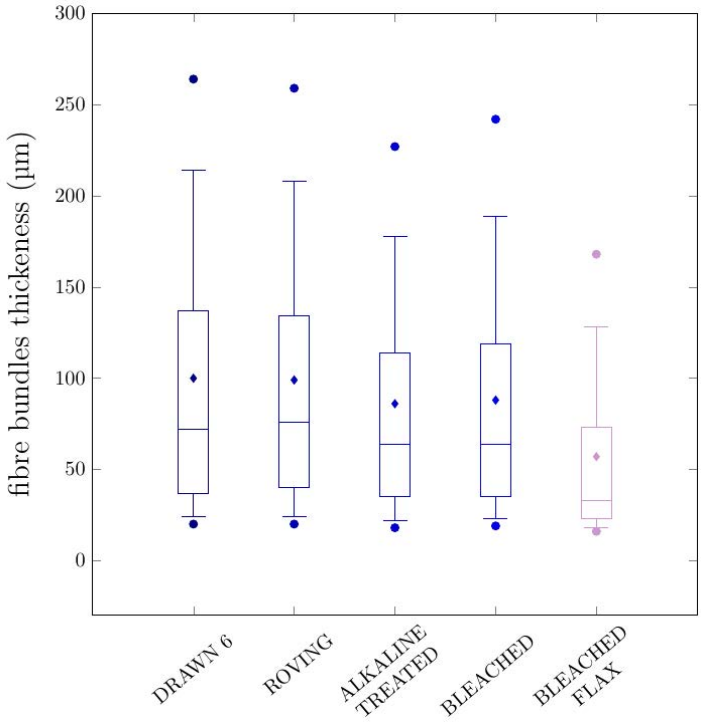


Figure 4.3: Thickness fibre bundles distribution before and after treatments.

The alkaline treatment participates to the decrease of the fibre bundles thickness. The addition of bleaching treatment did not show any size reduction. The alkaline treatment reduces the thickness of the fibres bundles although the hemp fibres bundles are still not as divided as the industrial bleached textile flax fibre bundles. The industrial bleaching was done with another degumming recipe.

4.1.2.2. Ring Spinning

For fashion textile sector, 24-Nm to 26-Nm yarns are generally targeted.

The roving threads were spun on a laboratory wet-spinning machine from Linimpianti company (Linificio e Canapificio Nazionale, Bergamo, Italy). Room temperature water was poured into the dedicated tray. The laboratory wet-spinning machine is presented in Figure 4.4.



Figure 4.4: Laboratory wet-spinning machine from Linimpianti.

Same parameters were kept as for spinning industrial textile flax. Hemp yarns between 40 to 60 tex (25 to 17-Nm) were spun.

4.1.2.3. Yarns Characterisations

Yarn Count

Prior to testing, the count was determined by calculating the ratio between mass and length. The count can be expressed in *tex* which is the mass in grams per 1000 metres (1 tex = 1 g/1000m). It can also be expressed in metric number, *Nm*, which is the yarn length in meters per gram (1 Nm = 1 m/g). The relation between the count in tex and the count in Nm is expressed in the equation (4-1).

$$1\ Nm = \frac{1000}{1\ tex} \tag{4-1}$$

The higher the Nm, the finer the yarns. The finer the yarns that can be spun without defects, the higher the value of the material.

Mechanical Properties

Mechanical properties of the yarns were assessed by tensile test. A semi-automatic tensile tester Tenso Lab 4 (Mesdan, Italy) (Figure 4.5) was used to realise the tensile tests according to UNE-EN ISO 2062:2009 standard.

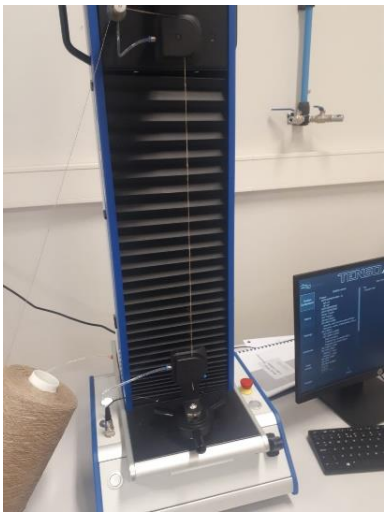


Figure 4.5: Tensile testing of yarn: TensoLab4 (Mesdan, Italy).

A load cell of 10 N with a pretension of 0.5 cN/tex, a test speed of 500 mm/min and a gauge length of 50 cm were used. A force-elongation curve was plotted, and the mechanical behaviour of the material was determined. The tensile strength at break, the elongation at break and work at rupture were collected. The tenacity was calculated according to the equation (4-2). Tests were carried out on at least 50 yarns.

$$tenacity\ (cN/tex) = \frac{maximum\ tensile\ force\ (cN)}{linear\ density\ (tex)} \quad (4\text{-}2)$$

Yarns Evenness

The yarn evenness corresponds to the variation of fineness along the length. It is commonly measured as the variation in mass per unit length along the yarn. It is an important property as it can influence many other properties. A yarn in which the variation in mass per unit length is small is said to be even, while the one in which this quantity exhibits large changes is considered as irregular. Variations are inevitable, due to the fundamental nature of the fibres and their arrangement (Slater, 1986).



Figure 4.6: MT Evenness tester unit (Mesdan, Italy).

An MT Evenness Tester (Mesdan, Italy) was used to realise the evenness tests (Figure 4.6). A test speed of 25 m/min was used on 25 m of yarn. An automatic calibration was done at the onset of each measurement. The data of the CV (%), the places thinner than 50%, thicker than 50% and the neps thicker than 280% are collected. Neps are entanglements of fibres. They are classified, like thin and thick places, as yarn faults or yarn defects and are strongly monitored during yarn quality control process in textile yarn spinning industries. The technical fibres that are not sufficiently divided (separated to each other) create a mass variation along the length of the yarn and lead to yarn unevenness. The unevenness degrades the fabric quality for commercial applications.

The quality of the yarns is presented in Table 4.2.

The hemp cultivated in Normandy can be processed in flax spinning system. Hemp only processed by mechanical treatment without any chemical treatment can be spun in 17 Nm yarns, although the yarns spun after alkaline treatment is finer, more even, and stronger than the yarns spun without treatment. It is observed that the strength of yarns increases with alkaline treatment, probably due to the removal of non-cellulosic component, but decrease with the addition of bleached treatment that might affect the cellulose components. The number of coarse irregularities decreased with bleaching, the neps were halved, but increasing the number of thin and thick irregularities. The greater the irregularity and the greater the number of irregularities, the greater the risk of yarn breakage in the next stages of textile design. In the subsequent stages of textile design, there is a greater chance of yarn breakage the more irregularities there are and the greater the irregularities. According to Yu (2013), yarns for textile applications spun with retted hemp fibres in China in a range of 12-32 Nm, have irregularity ranging between 32 and 45 CV% and a tenacity ranging from 17 cN/tex to 27 cN/tex *(Yu, 2013)*. The yarns manufactured in this work fit into the expected properties ranges.

Moreover, preliminary trials were carried at industrial scale out with Safilin company (Safilin, Sailly-sur-la-lys, France). and bleached yarns of Nm 18 were industrially spun and are useable for textile designer.

Table 4.2: Quality of untreated and treated hemp yarns and flax yarn.

Yarns	Count (tex)	Count (Nm)	Tenacity (cN/tex)	Maximum force (cN)	Elongation at maximum force (%)	Irregularity (CV%)	Thins (-50%/km)	Thicks (+50%/km)	Neps (+ 280%/km)
Untreated hemp	56	17	12.20 ± 3.74	683 ± 209	1.47 ± 0.41	51.27	4,469.3	558.7	1,037.5
Alkaline hemp	42	23	19.54 ± 5.06	821 ± 212	1.80 ± 0.41	39.7	3,511.6	439.0	1,436.6
Alkaline + Bleached hemp	41	24	16.56 ± 5.88	679 ± 241	1.56 ± 0.50	40.5	3,910.6	718.3	478.9
Alkaline + Bleached flax	36	28	27.95 ± 5.91	894 ± 189	2.06 ± 0.31	31.1	2,154.83	39.9	1,077.4

4.2. From the Non-Aligned Fibres to the Knitted Fabrics

4.2.1. Introduction

4.2.1.1. Context

In the precedent section, long line hemp fibres were used to produce fine yarns. Those fibres come from retted stems and their transformation process is following a scutching-hackling system combined with wet-spinning based on flax model. During the scutching part, about half of the fibres fall into tows. Those fibres are good quality dew-retted. Dew-retting means that plants are left spread out in the field and micro-organisms from soil partially degrade the substances that linked the bast fibres together and to the woody core of the plant stem. A good quality retting favours the decortication and the refinement ability of the fibres bundles. However, those tows possess very variable lengths and it is therefore difficult to spin them using the classic long fibres way by wet-spinning machine. Another suggested way to valorise those fibres is rotor-spinning in the frame of a blended yarn. Rotor-spinning is a technique that forms yarns without using spindles and offers advantages such as high production speed and cost effectiveness. The valorisation of those by-product fibres is possible in a blend with other types of natural or semi-natural fibres. An appropriate composed blend leads to the production of yarns suitable for knitting or weaving and those type of blended yarns are in increase demand on the clothing market. In the Institute of Textile Research and Industrial Cooperation of Terrassa (INTEXTER), a new approach of transformation of lignocellulosic wastes or by-products was recently developed and this method was patented (Patent No. 23 382 311.1).

In addition to hemp fibres, it was decided to study linseed flax fibres (Figure 4.7). The production of linseed flax production reached more than 37,000 ha in France in 2021 *(Terres Inovia, 2022)*.



Figure 4.7: Linseed flax field at seed maturity.

The plants are grown for the seeds and the fibres are mostly used for low added-value product or even burnt in some countries. Due to the lower stem length, the fibres cannot be extracted using the scutching approach. Yet, linseed flax fibres may have huge potential in different areas. Ouagne et al. reported that the fibre content in linseed stems varies between 37% and 40% *(Ouagne et al., 2017)*. Some authors those recent years, transformed linseed flax fibres into yarns for geotextile applications *(Khan et al., 2022).* However, it is suggested

that the optimisation of the dew-retting process of linseed flax could lead to wider application *(Bar et al., 2022)*. In this study, the linseed flax fibres will aim to be transformed into yarns dedicated to garment or housing textile applications.

4.2.1.2. The Modification of Fibres

The use of open-end rotor spinning technologies involves the modification of flax and hemp fibres. It implies processes causing the shortening and the division of the technical fibres. The fibres receive mechanical and chemical treatments. The mechanical treatments divide the fibres bundles by stretching and combing. A force is applied to separate the fibres bundles and gradually make a homogenous and fine sliver. The speed and gauge length are adapted to the material morphology and the final application, in other words the final count of the yarn. The chemical treatment, also called degumming, removes in large proportion lignin remaining from the fibres bundles and improves the division of the fibres. Hemp and flax fibres are build-up of a network consisting mainly of cellulose linked to hemicellulose and lignin. A high cellulose content gives the fibres high quality. An important amount of lignin is present in the middle lamellae of fibre bundles. Several methods for the elimination of non-cellulosic components exist. We will focus on the chemical degumming: alkaline and oxidising treatments are the most used chemical treatment. Alkaline is commonly done with sodium hydroxide (NaOH) solution. This treatment aims to remove hydrogen bonding in the network structure as explained by Figure 4.8 and Figure 4.9.

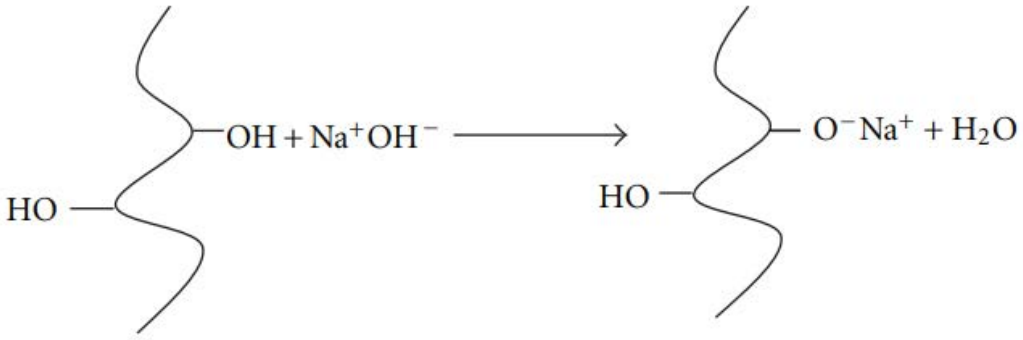


Figure 4.8: Alkali treatment reaction of cellulose-based fibres with NaOH from (Williams et al., 2011).

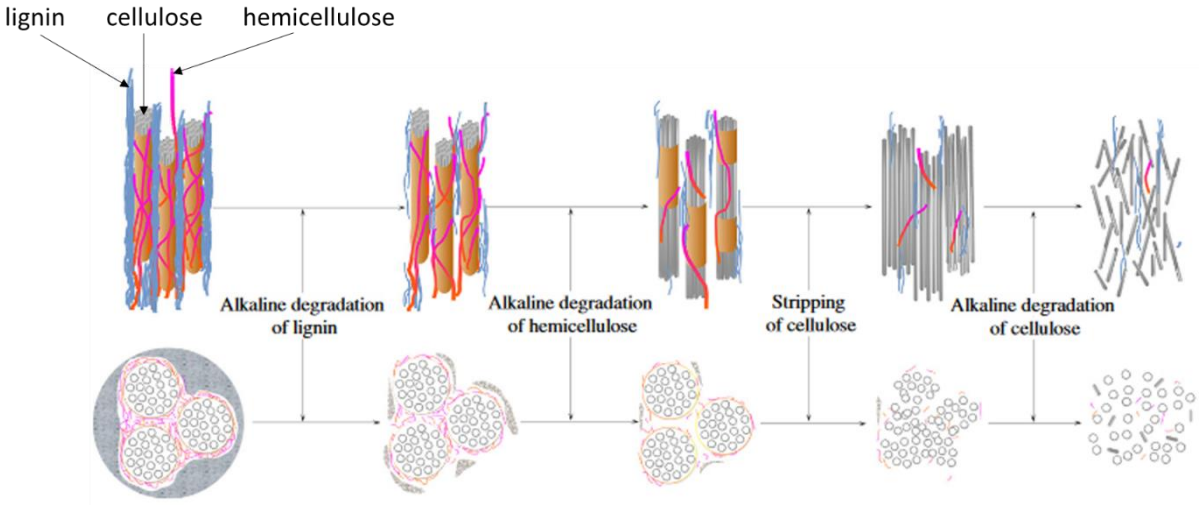


Figure 4.9: Scheme of natural fibre alkaline degradation process from (Wei and Meyer, 2015).

After alkaline treatment, fibres are subjected to an oxidising process to remove the remaining lignin and reach a higher level of fibre division, although its principal aim is whiteness achievement. The most commonly used oxidising agents for textiles are hydrogen peroxide (H_2O_2), sodium hypochlorite (NaClO), sodium chlorite ($NaClO_2$) and peracetic acid ($C_2H_4O_3$). We will focus on oxidising with hydrogen peroxide, which is a strong oxidizing agent and an environmentally friendly product as it decomposes to O_2 and H_2O. The oxidising treatment is performed at high temperatures under alkaline conditions using NaOH with wetting agents and stabilizers or activators if needed. In alkaline media, the equilibrium of H_2O_2 shifts to the formation of perhydroxyl anions (HOO^-) (Figure 4.10). It is the principal active species in peroxide bleaching as it will react with the chromophore groups of lignin *(Hobbs and Abbot, 1991).* Indeed, in the case of lignocellulosic fibres, the colour of the fibres is due to the lignin component. The strong nucleophile (HOO^-) will attack the unsaturated or conjugated carbonyl groups and lead to the permanent removal of most of the chromophoric groups present in the lignin molecule.

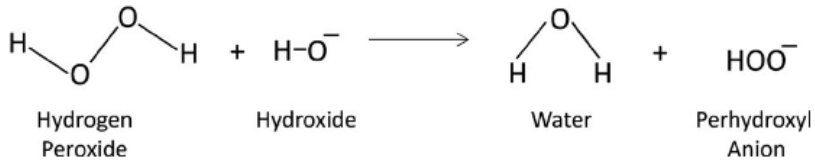


Figure 4.10: Chemical reaction mechanism of H_2O_2 in alkaline media.

During the process, another reaction is happening and corresponds to the decomposition of hydrogen peroxide (H_2O_2) in water (H_2O) and dioxygen (O_2) (Figure 4.11). The rate of hydrogen peroxide decomposition increases with the increase of temperature, concentration and pH *(Sharma and Sumere, 1992)*.

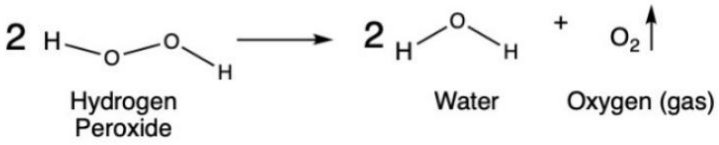


Figure 4.11: Decomposition of hydrogen peroxide.

This reaction needs to be limited. At high temperature, to slow down the rapid decomposition of hydrogen peroxide, a stabiliser can be applied, such as sodium silicate or phosphonic acids, or more environmentally friendly, oligomeric acrylic acid. By working at lower temperature, the decomposition of hydrogen peroxide can be limited but an activator should be used to promote the reaction in Figure 4.10.

4.2.1.3. The Objectives

The purpose of this study is to investigate the processability of by-product materials to form yarns by open-end rotor spinning. The aim is to produce yarns with a high content of by-products materials (higher than 50%). First, the effect of chemical degumming on several types of fibres is studied at laboratory-scale. The research group previously carried out studies in this field with different ligno-cellulosic fibrous resources mainly for paper applications (Mijas et al., 2022, 2021) and it was decided to use two recipes of degumming treatments. After these preliminary treatments, the most promising treatment was further investigated and up-scaled at semi-industrial scale. A first part details the tests and the characterization on fibres and yarns at laboratory scale and a second part describes the up-scaling process and the characterizations on the spun yarns and knitted fabrics. Finally, the results are discussed.

4.2.2. Laboratory Scale Experiments

The methodology carried out at the laboratory-scale is outlined in the flow-chart illustrated in Figure 4.12.

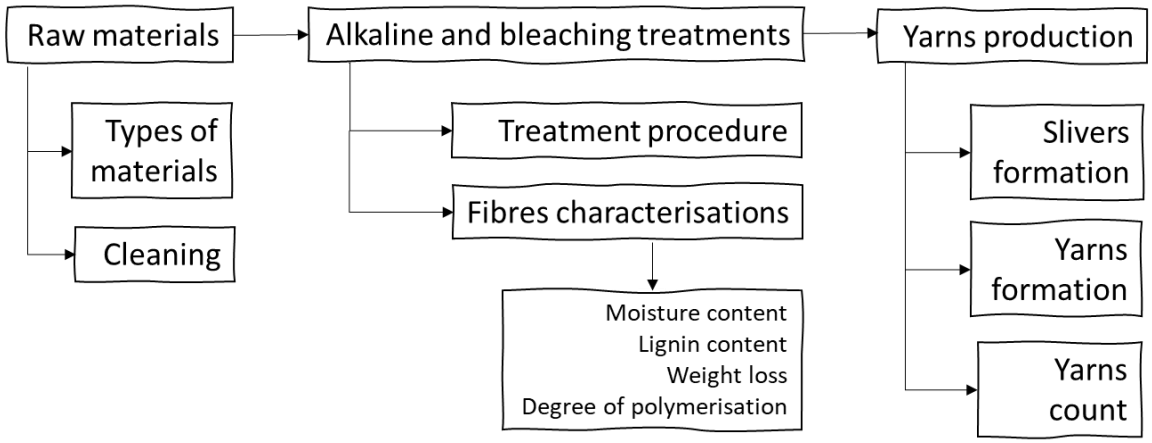


Figure 4.12: Flow chart of the methodology of open-end rotor spinning of fibres at laboratory-scale.

4.2.2.1. The Raw Materials

Types of Materials

Four types of material were studied:

- **SAMPLE A**: Hemp fibres that are used in paper industry. They were unretted fibres, extracted by decortication leading to a blend of medium-short fibres and a high presence of shives.

- **SAMPLE B**: Hemp fibres that were retted, and had medium to long length.

- **SAMPLE C**: Hemp fibres that were retted, had medium length and are not processable with the traditional wet-spinning line; received from Hemp-Act company (Lacapelle-Marival, France)

- **SAMPLE D**: Linseed flax fibres that were mid-retted, and were from the waste products of the harvest of linseeds from South-west of France; Fibres extraction performed at Hemp-Act company (Lacapelle-Marival, France)
-

Cleaning Procedure

The first step was to clean the material from dust and other surface residues. Fibres were washed with a 1.7 g/L surfactant (Hostapal UH Liq., Clariant), mixture of anionic-non-ionic surfactant, with a liquor ratio 1:30 (w/V) at (70 ± 2) °C for 10 min and rinsed three times with distilled water at room temperature for 5 min. Finally, hemp fibres were dried at room temperature.

4.2.2.2. Alkaline and Bleaching Treatments

The methodology carried out for alkaline and bleaching treatments is summarized in the flow-chart illustrated in Figure 4.13.

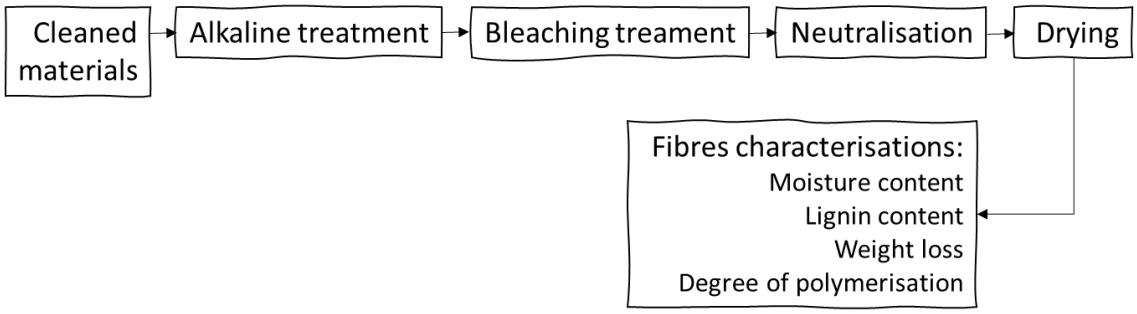


Figure 4.13: Flow chart of the chemical processes at laboratory-scale.

Treatments Procedure

Treatments at high temperature and alkaline medium were carried out to ensure the elimination of the greatest amount of lignin. Oxidising treatments were performed on hemp fibres as a complementary treatment of alkaline treatment for the delignification of hemp and to achieve fibres with a higher degree of whiteness.

Alkaline and oxidising treatments were performed successively in a Linitest (Heraus Hanau, Germany), illustrated in Figure 4.14.



Figure 4.14: Laboratory dyeing and wet finishing machine, Linitest (Heraus Hanau, Germany).

A concentration of 1.5 g/L of a surfactant (mixture of anionic-non-ionic surfactant, Hostapal DTC, Clariant), and a liquor ratio of 1:20 (w/V) were used. Liquor was prepared and placed in the vessels along with the fibres.

For the alkaline treatment, two concentrations of NaOH were studied: 0.4 mol/L and 0.2 mol/L. The bath temperature was set at 80 °C and the time of reaction was 2 hours. The parameters were chosen according to precedent studies from *(Mijas, 2022)*. Between the alkaline treatment and the oxidising treatment, the sample was rinsed with distilled water three times.

For the oxidising treatments, a concentration of 3 mL/L of H_2O_2 50% (w/V), 1 mL/L of NaOH 50% (w/V), 2 mL/L of an activator (Adrapur AIO-L (Adrasa)) were chosen, the bath temperature was set at 60 °C and the time of reaction was established at 30 minutes according to precedent studies from *(Mijas, 2022).*

Once the reaction time elapsed, the sample was rinsed with distilled water, neutralised with acetic acid (6 g/L), rinsed with distilled water three times and dried at room temperature.

Fibres Characterisations

Moisture content

Specimens were grinded into 2 mm length with a blender (Moulinex, France). The amount of (1.0 ± 0.3) g of grinded test specimen was weighted and place in an oven at (105 ± 3) °C overnight. The test specimen was weighted again after cooling in a desiccator for 2 hours. The moisture factor was calculating with the following equation (4-3).

$$f\ (\%) = \frac{dry\ hemp\ mass}{wet\ hemp\ mass} \quad (4\text{-}3)$$

Lignin content

Lignin content (%) in hemp fibres after the chemical treatments was calculated based on Tappi Standard T 222 om-2015 "Acid-insoluble lignin in wood and pulp". The obtained lignin content is not representative of the original lignin but it constitutes the major mass proportion of the lignin content.

Sulphuric acid, 72% H_2SO_4 solution, (24 ± 0.1) N (density 1,634 g/cm^3) was prepared as follows: 665 mL of concentrated H_2SO_4 (96%) (Scharlau, Spain) were poured into 300 mL of water, and after cooling filled up to 1000 mL. The strength was adjusted by measuring density using a densimeter. The acid solution was cooled in a refrigerator between 10 and 15 °C before using.

Procedure:

- Hemp fibres were grinded into 2 mm length with a blender (Moulinex, France).
- (2.0 ± 0.1) g of grinded test specimen were weighed and placed in a 100-mL beaker. Two specimens were done for each batch.
- 40.0 mL of cold 72% H_2SO_4 solution were added gradually to the test specimens while stirring with glass stick. The beaker was kept in a cold bath during dispersion of the material. After the specimen was dispersed, the beaker was covered and kept in a bath at (20 ± 1) °C for 2 hours. The material was stirred every 15 minutes to ensure complete solution.
- The solution was transferred to a 2000 mL Erlenmeyer flask and a dilution with water was made to 3% concentration of H_2SO_4 (final volume of 1,540 mL). The solution was kept boiling for 4 hours, maintaining constant volume by using a reflux condenser.
- The solution was left to decant overnight, keeping the flask in an 45° inclined position.
- The supernatant solution and the insoluble material (lignin) were filtered through a filtering crucible containing a disc of a glass fibre paper (Whatman) and rinsed with hot water.
- The crucible with filter and lignin was dried at (105 ± 3) °C overnight. Then, it was cooled in a desiccator and weighed.

For each specimen, the lignin content was calculated following equation (4-4).

$$Lignin\ (\%) = \frac{weigth\ of\ dried\ residual\ lignin\ (g)}{weight\ of\ dried\ specimen\ (g)} \times 100 \quad (4\text{-}4)$$

Weight loss

The test specimens were conditioned at a room temperature of (20 ± 2) °C and (65 ± 4)% RH for 24 hours prior the treatment and after the treatment and were weighted. The weight loss was calculated with equation (4-5).

$$Mass\ loss\ (\%) = \frac{mass\ of\ the\ treated\ material\ (g)}{mass\ of\ the\ initial\ material\ (g)} \times 100 \quad (4\text{-}5)$$

Degree of polymerization (DP)

Bleaching is a technique that oxidises the fibres and can create damages. During bleaching the cellulose chain can be negatively modified. The state of the cellulose chain can be observed by the measurement of the degree of polymerisation (DP). The DP was determined according to the AFNOR Standard NF T12-005 with a cupriethylenediamine solution. DP was calculated with the average viscosity. It is estimated from the intrinsic viscosity of the dissolution of the material in a 0.5 mol/L cupriethylenediamine hydroxide solution by application of the Mark-Houwink-Sakurada (MHS) equation, presented in equation (4-6).

$$\eta = K\overline{M_v^{\alpha}} = K'\overline{DP_v^{\alpha}} \quad (4\text{-}6)$$

with η is the intrinsic viscosity, M is the molecular weight, α, K and K' are constants dependent on the polymer-solvent system.

Procedure:

- 20-25 mg of grinded specimens were poured into a 25-mL flask. Four drops of the Hostapal UH liq surfactant (same surfactant as mentioned in section 4.2.2.1) and five glass beads were added, and the flask was shaken.
- 35 mL of 0.5 mol/L cupriethylenediamine hydroxide solution (Scharlau, Spain) was poured, so then when closed with a cap containing a glass capillary, the liquid entirely replaced the air without any remaining bubbles and ascended the capillary tube. The samples were shaken mechanically for 3 hours.
- 10 mL of the solution were then poured into the viscosimeter and placed in a bath at (28 ± 1) °C for 10 minutes. The solution was risen to the top of the viscosimeter by a partial vacuum and then freely drained out; when the meniscus passed the upper mark, the timer is started until the solution reached the lower mark. This time correspond to the efflux time. Five successive measurements of the efflux time were carried out.

The specific viscosity η_{sp} can be expressed as a function of the efflux time (equation (4-7)).

$$\eta_{sp} = \frac{\eta}{\eta_0} - 1 = \frac{\eta - \eta_0}{\eta_0} = \frac{t - t_0}{t_0} \quad (4\text{-}7)$$

With η_0 the viscosity of the solvent, η measured viscosity of polymer solution at known concentration *c*. The intrinsic viscosity was calculating by applying Martin's formula with k = 0.14 (equation (4-8)).

$$\log \eta_{sp} = \log(c.\eta + k.c.\eta) \quad (4\text{-}8)$$

Tables given in standards were used to determine the values $c.\eta$ and $c.\overline{DP_v}$ considering *K'* = 7.5 x 10^{-3} and α = 1 *(Evans and Wallis, 1989)*.

4.2.2.3. Yarns Production

Slivers Formation

For the use of an open-end rotor spinning system, the fibres were too long and need to be cut. Hence, before making a sliver, hemp fibres were cut with a flat manual guillotine into 4 to 5 cm length fibres. During the sliver formation, mechanical treatment will be applied and might again reduce the size of the fibres. If the fibres are too short, they cannot be spun into yarns anymore. Hence, the size of cutting was chosen according to the fibres length requirements of rotor spinning, which is around 3 cm to 3.5 cm. After cutting, an emulsion was spread on fibres to limit the frictions. The emulsion was formulated in the laboratory containing 5.8 g/L of hemp seed oil (Marnys, Spain), 0.7 g/L of sodium lauryl sulfate (anionic surfactant) (Merck, Germany), 0.7 g/L of Tween 20 (non-ionic surfactant) (Merck), 0.7 g/L of Synperonic A-7 (non-ionic surfactant) (Croda, UK) and distilled water. For each 5 g of hemp, 10 mL of emulsion was applied and left to dry at room temperature for at least 12 h.

Fibres were then blended with another type fibres to give cohesion and deformability properties to the material. Blends of cotton and hemp or flax were made with the following mass proportions:

- Cotton 60% / hemp or flax 40% (w/w)
- Cotton 50% / hemp or flax 50% (w/w)
- Cotton 20% / hemp or flax 80% (w/w)

A quality control machine for cotton fibres, Uster-MDTA 3 (Textechno, Germany) presented in Figure 4.15, consisting in a fibre opening unit and a rotor-ring was used to make slivers of 1-meter circumference. The materials were passed 3 times through the Uster-MDTA 3 machine to obtain a homogenous sliver *(Das et al., 2004).*



Figure 4.15: Micro dust and trash analyser, Uster-MDTA 3 (Textechno, Germany), used as laboratory sliver forming machine.

One of the essential requirements of blended yarn is the homogeneity in blending their components throughout the length of yarns spun and the uniform distribution of the individual fibre component in the cross-section. The used method aims to meet these needs.

Yarns Formation

Yarns were spun with a rotor-spinning machine. Yarns were spun on a modified Autocoro SE11 (Schlafhorst, Saurer, Germany) open-end rotor machine (Figure 4.16).



Figure 4.16: The open-end rotor spinning machine.

Slivers of fibres of approximately 5 g/m directly fed the machine through the feeding roller. The fibres come into contact with an opening roller covered with pins, which combs out individual fibres and creates a thin net of fibres. These fibres are then deposited on the outer edge of the rotor. The 56-mm diameter T-shape rotor spins rapidly, imparting twist to the fibres. Centrifugal force and partial vacuum in the rotor cause the fibres to be continuously taken out and incorporate into the rapidly spinning "open end" of a preformed yarn that extends out of the draw-off tube and wound onto the package. A scheme of the open-end rotor system is presented in Figure 4.17.

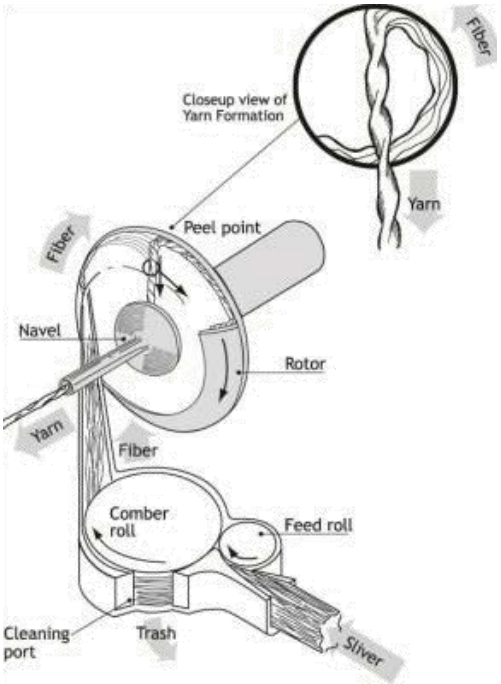


Figure 4.17: Scheme of open-end rotor system from textilelearner.net

Yarns were spun by adjusting the rotor speed, the alimentation speed and the production speed according to tables from previous settlements. When a single sliver is too light to create a yarn, slivers might be doubled to increase the feed.

Yarns Characterisations

Yarns were characterized by measuring their counts. It was determined by enrolling 50 m of yarns around a wrap reel and weighed. The count in *tex* was obtained by calculating the ratio between mass and length, with units in g/1000m.

4.2.3. Semi-Industrial Scale Experiments

After the treatment at laboratory scale the most promising treatment was produced at semi-industrial scale. The up scaling generally leads to more homogeneity and higher quality products.

4.2.3.1. The Raw Materials

At semi-industrial scale, only the SAMPLE C and SAMPLE D were studied.

4.2.3.2. Alkaline and Bleaching Treatments

Fibres were processed at a semi-industrial scale by choosing the most appropriate conditions from lab scale. The treatment occurred in a stainless-steel reactor made of a 110 m^3-volume with liquid flow. (5.0 ± 0.1) kg of test specimen are enclosed in a pierced basket, in respect with the liquor ratio studied previously 1:20 (w/V). The alkaline mixture is prepared in a stainless-steel tank with a minimum of 160 L required covering the pierced basket and the heating resistance. The liquor continuously circulates through the basket and the tank. Experimental conditions for bleaching treatments were the same as lab-scale, described in section 4.2.2.2. After treatment, fibres were rinsed with water and neutralised with acetic acid and water three times. Tap water was used at semi-industrial scale instead of distilled water. After treatment, the fibres were spin-dried to remove the excess of water and were dried at ambient temperature or in an oven at 60 °C. The fibres dried faster while spreading them at ambient temperature than in the oven (without any relative humidity control), in addition it dries without large changes in temperature that could impact fibres properties and it is lower energy consuming. This type of drying was adapted for the amount of treated material in this study, however, for larger quantities another solution might be needed.

4.2.3.3. Yarns Production

Veil Formation

To fit to the open-end rotor spinning system, the fibres were cut. The large quantity of fibres requires a suitable automatic tool for cutting instead of the manual guillotine used at laboratory-scale. Fibres were cut into short length pieces with a recycle textile cutting machine. The length of the pieces was heterogeneous in a range of 3 cm to 5 cm. Fibres are then blended with other fibres to form yarns. This time, Lyocell was used instead of cotton. Lyocell is a form of regenerated cellulose made by dissolving pulp. It was chosen to calculate the actual amount of each material in the final yarn. By chemical analysis it is possible to only dissolve the cellulose of Lyocell and obtain the weight of the remaining flax or hemp. This technique is not possible with a cotton blend.

Fibres were blended with Lyocell in two different proportions:

- Lyocell 50% / Fibres 50 % (w/w)
- Lyocell 20% / Fibres 80% (w/w)

A revolving flat card (Platts, Werneth, Oldham, England) was used to blend the fibres and produce a strand (Figure 4.18). A sheet of natural fibres was spread out on the preparation bench of the machine and Lyocell fibres were spread on it. The revolving flat card is stretching out the fibres and the fibres arein a less tangled arrangement. It produces a strand mostly free of impurities, as most of the remaining shives or woody part are fall out.

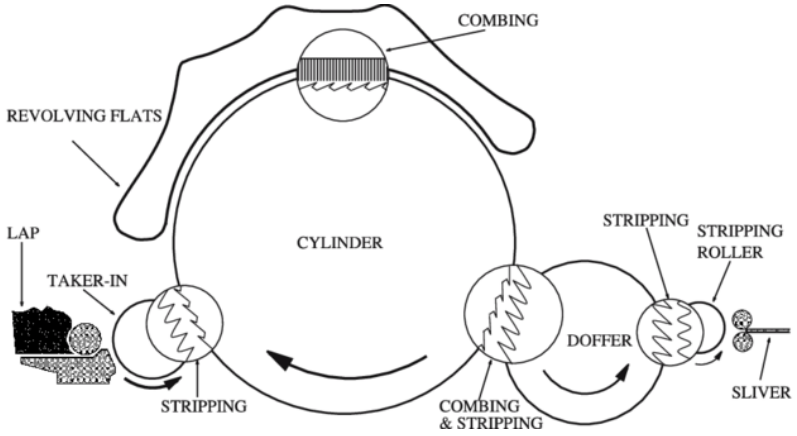


Figure 4.18: The transfer of fibres in the carding machine, from (Lee and Ockendon, 2006).

Fibres essentially encounter hooks distributed on rapidly rotating cylinders and quasi-stationary flat plates. The first of the three cylinders is called the taker-in, and this introduces the initially entangled fibres, named lap in Figure 4.18, into the carding machine. The hooked flat plates adjacent to the largest cylinder act to break down and turn bunch of fibres into individual fibres. This leads to the formation of a smooth coherent strand, called the sliver, by the transfer of individual fibres from the main cylinder to the removal cylinder, known as the doffer. The physical properties of the further resulting yarn depend critically on the formation of cohesive structures, web, during the carding process.

An initial blend of (50 ± 0.5) g was used. After the first passage, the strand is doubled and passed twice more through by the card rendering the fibres in the final strand more cohesive and parallel and enhancing the regularity of the thickness of the strand.

Slivers Formation

A draw frame machine (Platts, Werneth, Oldham, England) is used to make the fibres into a sliver much more parallel and the sliver, itself, much more regular in thickness. In our case, the draw frame was also used to adjust the linear mass of the strand to the desired one for spinning. A group of chosen number of strands is assembled and passed through pairs of rollers with the adequate roller speed.

The operation was repeated three times with a gradual setting of drafting. The first passage, the strand from the card is processed. The second passage four slivers were fed together. The final passages four, five or six slivers were fed together depending on their thickness and knowing that the final sliver count should be comprised between 3.0 to 5.0 g/m to correctly fit to the open-end spinning machine. The distance between the rollers can be adjusted according to the length of the processed fibres. The distance between the rollers was as follows: d1: 4.19 cm, d2: 4.32 cm, d3: 4.83 cm, as mentioned in Figure 4.19. They were chosen based on previous studies on jute fibres.

The drafting is calculated according to the equation (4-9).

$$Drafting\ (\%) = \frac{Delivery\ speed}{Feed\ speed} \quad (4\text{-}9)$$

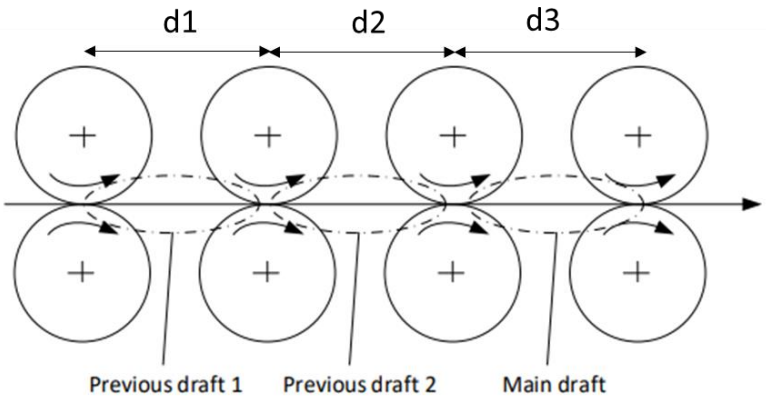


Figure 4.19: Scheme of rollers and drafts of the draw frame machine.

The parameters of draft are summarised in the Table 4.3.

Table 4.3: Draft during the process of slivers formation.

	1st Step	2nd Step	3rd Step
Gear (Z)	117	87	40
Previous draft I (-)	1.7	1.7	1.7
Previous draft II (-)	1.03	1.03	1.03
Main draft (-)	5.45	4.05	1.86
Total draft (-)	9.54	7.1	3.26
Delivery speed (m/min)	18.8	18.8	18.8

Slivers containing hemp or linseed flax were made without any difficulties without any sizing.

Yarns formation

Yarns were spun according to the same methods previously mentioned in the section 4.1.2.

Yarns characterisations

Microscopic characterisations

Microscopic observations were made under an optical microscope (Olympus PMG3-F3, France) with a magnification x10.

Mechanical properties

Mechanical properties of the yarns were assessed by tensile test. Prior to testing, yarns were conditioned at (65 ± 4)% R.H. and (20 ± 2) °C for 24 hours. An automatic Tensile Tester Statimat ME (Textechno, Germany); presented in Figure 4.20, was used to realise the tensile tests according to UNE-EN ISO 2062:2009 standard.



Figure 4.20: Charging cells of the automatic Tensile Tester Statimat ME (Textechno).

A charge cell of 10 N with a pretension of 0.5 cN/tex, a test speed of 500 mm/min and a gauge length of 50 cm were used. A force-elongation curve was plotted, as shown in Figure 4.21, and the mechanical behaviour of the material was determined. The tenacity, the breaking strength, the tensile strength at break, the elongation at break and work at rupture were determined. Tenacity was calculated according to the equation (4-10). Tests were carried out on at least 50 threads of one yarn sample.

$$tenacity\ (cN/tex) = \frac{maximum\ tensile\ force\ (cN)}{linear\ density\ (tex)} \qquad (4\text{-}10)$$

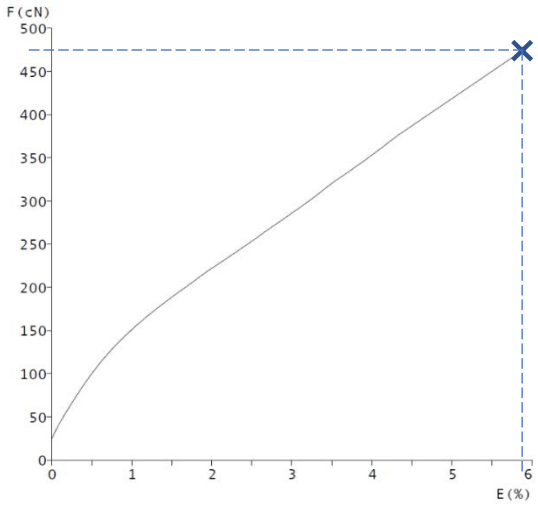


Figure 4.21: Average force/elongation diagram. A: Force at break. B: Elongation at break.

Final yarn composition

During the entire transformation process and especially during yarns open-end spinning, some fibres were lost falling into dust. These losses impacted the composition ratio of the final yarn. By blending the material with Lyocell it is possible to determine the final composition through chemical analysis. The determination of the ratio Lyocell/natural fibres was made by dissolving the Lyocell cellulose in order to weight the residual flax/hemp. It was made by following the European standard: AENOR. UNE-EN ISO 1833-22:2022 Textiles - Quantitative chemical analysis. Part 22: Mixtures of viscose or certain types of cupro or modal or Lyocell with flax fibres (method using formic acid and zinc chloride).

The first part consists in a pretreatment for removing non cellulosic components.

Procedure:

- (1.2 ± 0.1) g of final yarn were cut in 2 mm pieces and placed in a 100-mL round flask. Two specimens were done for each batch.
- A solution of 1.5 mol/L sodium hydroxide was added to the flask up to 100 mL. Liquor ratio is 1/100.
- The material was boiled for 1 hour and then rinsed 5 times with distilled water.
- The material was neutralised with 0.1 mol/L acetic acid for 10 minutes and then filtered with acetic acid and distilled water. The sample is dried, cooled down in a desiccator and weighed.

Second part consists of removing Lyocell cellulose.

Procedure:

- A formic acid / zinc chloride solution was prepared by dissolving 20 g of anhydrous zinc chloride (≥ 98 %) and 68 g of anhydrous formic acid and made up to 100 g with distilled water.
- An ammonia solution was prepared by diluting 20 mL of concentrated ammonia solution (ρ = 0.880 g/mL at 20 °C) in 1 L of distilled water.
- (1.0 ± 0.2) g of pre-treated material were added to a round bottom flask preheated to 70 °C and 100 mL of a formic acid and zinc chloride solution pre-heated to 70 °C was added to flask. The sample was maintained at 70 °C for 20 minutes and shook twice, at 5 minutes and 15 minutes.
- The solution was filtered through a filtering crucible and with 20 mL of the formic acid and zinc chloride solution at 70 °C and with distilled water at 70 °C.
- The crucible was placed standing in a 250-mL beaker filled with 100 mL of cold ammoniac solution for 10 minutes and material was moved with a glass rod. The crucible was dried by suction.
- The crucible was rinsed with cold distilled water and dried overnight at (105 ± 3) °C. Then, it was cooled in a desiccator and weighed.

For each sample, the percentage of mass loss during pretreatment is calculated using formula in equation (4-11).

$$P_s = \frac{m_1 - m_2}{m_1} \times 100 \quad (4\text{-}11)$$

With m_1 the dry mass of the tested specimen before the pretreatment and m_2 the dry mass of tested specimen after the pretreatment.

The dry mass of the mixture after transfer corrected to its initial dry mass before pretreatment is calculated by equation (4-12).

$$M = \frac{100.m_3}{100 - P_s} \quad (4\text{-}12)$$

With m_3 the initial dry mass used for the second treatment

The dry masses of Lyocell and pretreated fibres are calculated using equations (4-13) and (4-14).

$$l = d_1(m_3 - d_2.m_4) \quad (4\text{-}13)$$

$$f = M - l \quad (4\text{-}14)$$

With d_1 the corrected factor for loss in mass of Lyocell during the pretreatment and d_2 is the corrected factor for loss in mass of bast fibres during the treatment with formic acid/ zinc chloride reagent.

The percentage of each component is calculated using equations (4-15) and (4-16).

$$L = \frac{l.\left(1 + \frac{t_{c1}}{100}\right)}{l.\left(1 + \frac{t_{c1}}{100}\right) + f.\left(1 + \frac{t_{c2}}{100}\right)} x100 \quad (4\text{-}15)$$

$$F = \frac{l.\left(1 + \frac{t_{c1}}{100}\right)}{l.\left(1 + \frac{t_{c1}}{100}\right) + f.\left(1 + \frac{t_{c2}}{100}\right)} x100 \quad (4\text{-}16)$$

With t_{c1} the percentage addition for moisture added to the dry mass of Lyocell (t_{c1} = 1.13); and t_{c2} the percentage addition for moisture added to the dry mass of pretreated flax and hemp bast fibres (t_{c2} = 1.12).

This method measures the proportion of each material blended with Lyocell through the cellulose contents and, however, it does not include the other component of hemp and flax such as hemicellulose or lignin. Thus, the final evaluated proportion of the material into the yarn is a bit under-estimated compared to the real one.

4.2.3.4. Fabric Production

A single-cylinder circular knitting machine, designed and assembled in INTEXTER from a simplified model of industrial socks machine was used for manufacturing fabrics. The machine is presented in Figure 4.22.



Figure 4.22: Circular lab-knitting machine.

It has a static creel, a gauge E23, a rotating cylinder with a diameter of 90 cm and 65 needles. Feed rate was adjusting automatically by the machine according to the characteristic of the yarn. Final product is weft knitted fabric as illustrated in Figure 4.23.

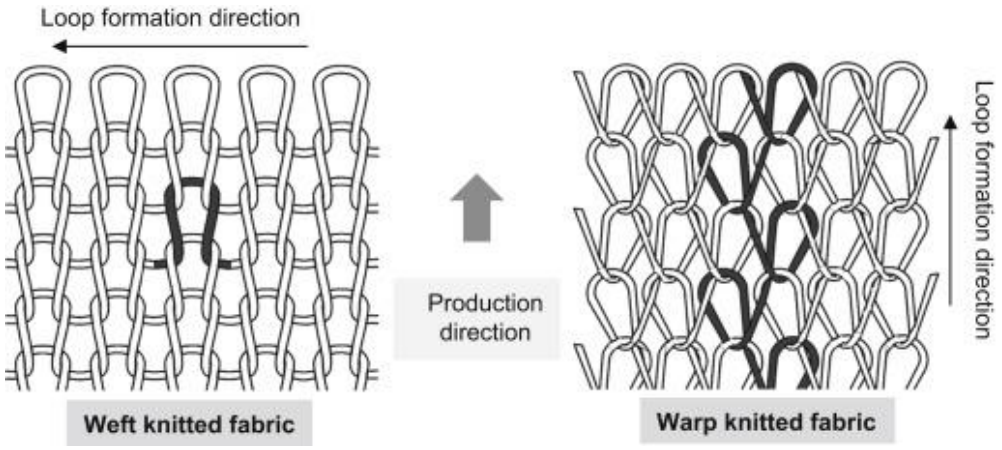


Figure 4.23: Structure of warp and weft knitted fabrics . (V. Schrank et al. 2017, Polymer Optical Fibres).

4.2.4. Results

4.2.4.1. Laboratory Scale

Degumming treatments were first studied at laboratory scale to observe the effect of the treatment on the fibres. The degumming treatments include first an alkaline part and secondly a bleaching part. Two different concentrations were tested during the alkaline treatment: NaOH 0.2 mol/L and NaOH 0.4 mol/L.

The Table 4.4 gathers the results of mass losses after treatments. The non-cellulosic components, mostly lignin, and remaining pectin, wax are expected to be removed using the treatment.

Table 4.4: Weight losses after alkaline and bleaching treatment.

Batches	Weight removed after treatment with NaOH 0.2 mol/L (%) (w/w)	Weight removed after treatment with NaOH 0.4 mol/L (%) (w/w)
SAMPLE A	20.2 ± 0.1	24.2 ± 0.2
SAMPLE B	15.1 ± 0.3	17.4 ± 0.4
SAMPLE C	13.9 ± 0.4	17.0 ± 0.3
SAMPLE D	20.5 ± 0.1	23.4 ± 0.1

The removed lignin has a main impact on the further steps of the process as it is largely responsible for the binding of the fibres and the size in length and in thickness of the fibres bundles. It was deeper studied by carrying out Klason tests. The Table 4.5 gather the content of lignin in the material before and after treatments.

Table 4.5: Lignin proportion in material before and after treatments.

		Treatment with NaOH 0.2 mol/L		Treatment with NaOH 0.4 mol/L	
Batches	Initial lignin content (%) (w/w)	Final lignin content treated 0.2 mol/L NaOH (%) (w/w)	Removed lignin (%) (w/w)	Final lignin content treated 0.4 mol/L NaOH (%) (w/w)	Removed lignin (%) (w/w)
SAMPLE A	7.2 ± 0.4	4.6 ± 0.3	37	3.6 ± 0.4	50
SAMPLE B	4.0 ± 0.1	2.6 ± 0.2	35	2.8 ± 0.3	29
SAMPLE C	4.4 ± 0.1	2.7 ± 0.2	38	3.3 ± 0.2	24
SAMPLE D	5.7 ± 0.6	4.1 ± 0.9	29	4.1 ± 0.7	28

The lignin content decreases with the alkaline treatment. It can be observed that increasing the concentration of NaOH during alkaline treatment has a low impact on the final lignin content in the material. The values at the two different concentrations are very close to each other and even a bit higher with the lower concentration for the retted hemps. This can be explained by the heterogeneity of the material. We can notice that linseed flax (SAMPLE D) has a higher initial amount of lignin than the retted hemp (SAMPLE B and C).

It is interesting to observe the effect of the treatment on the cellulose. Cellulose is the major component of hemp and flax (55%-85%) *(Bourmaud et al., 2018)* and its polymeric state has an impact on the mechanical properties of the material. The degree of polymerisation is a good indicator of the polymer chain properties. The results of the DP measurement before the treatments and after the treatments are represented in Figure 4.24.

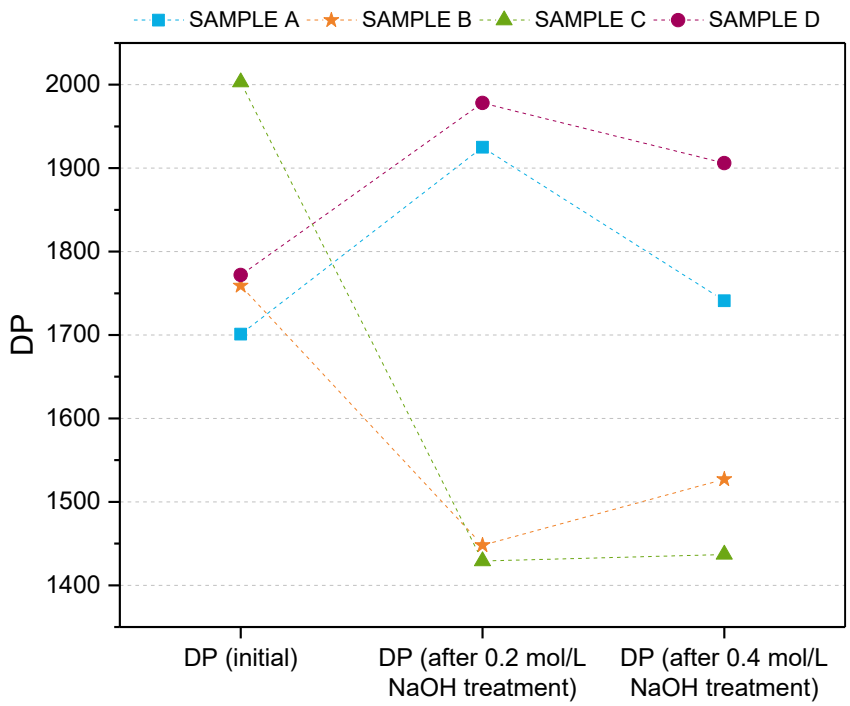


Figure 4.24: Degree of polymerisation (DP) according to AFNOR Standard NF T12-005 method, of each material before and after treatment.

The average DP value of cellulose is altered by the presence of hemicellulose and lignin in the solution of cupriethylenediamine *(Hallac and Ragauskas, 2011; Kaminska, 1996).* Lignin contains smaller polymer chains than cellulose. Lignin are partly removed during the alkaline treatment and consequently for the samples with the highest initial lignin content (SAMPLE A: unretted hemp and SAMPLE D: mid-retted flax), the DP values is increasing after a treatment with 0.2 mol/L NaOH. The short polymers chains are supposed to be in lower proportion in the treated material than the initial one. When the alkaline concentration is higher (0.4 mol/L NaOH) the cellulose is supposed to be affected, the cellulose polymers chains are slightly damaged and the DP decreases. For the samples with the lowest initial lignin content (SAMPLE B and C: retted hemps) the cellulose is affected, damaged, and the average DP is lightly altered.

The values of DP show that cellulose was affected but they still reach value higher than 1,400 which is supposed to be high enough to manufacture a yarn in this case. Huge part of lignin was removed reaching a final concentration of approximately 3% in retted hemp (SAMPLES B and C) and 4% in mid-retted flax (SAMPLE D) and unretted hemp (SAMPLE A).

Treated fibres were blended with cotton to form a sliver in the three different proportions (Figure 4.25):

- cotton 60% / hemp or flax 40% (w/w),
- cotton 50% / hemp or flax 50% (w/w),
- cotton 20% / hemp or flax 80% (w/w).



Figure 4.25: Slivers made of blend of cotton and by-products.

The slivers were spun with the open-end spinning machine. During spinning a huge proportion of the material was transformed in waste by the opening roller. Consequently, the final counts are different from the desired ones. The Table 4.6 gathers for the different spun yarn the different of fibres and the desired and measured counts.

By visual observations of the rejected fibres, it was accepted that they were hemp or flax fibres predominantly. Assuming that all the material lost during spinning was these fibres and not cotton, we recalculated the proportion of the final yarn. Finally, 70 tex yarn was spun with 76% of linseed flax and 54 tex yarn was spun with 71% of hemp. Those trials were made to test the ability of the sliver to be spun.

Table 4.6: Examples of yarns count in tex spun wth open-end process.

Material	Proportion (cotton/hemp)	0.2 mol/L NaOH		0.4 mol/L NaOH	
		Nominal count	Experimental count	Nominal count	Experimental count
SAMPLE A	60/40	81	56	77	44
	50/50	81	51	72	46
SAMPLE B	60/40	60	49	55	36
	50/50	80	58	81	56
	20/80	-	-	79	57
SAMPLE C	60/40	81	60	79	32
	50/50	60	47	81	45
	20/80	-	-	78	54
SAMPLE D	60/40	50	40	80	65
	50/50	50	42	79	65
	20/80	80	66	81	70

Yarns were not spun with the sample B and C in proportion cotton/hemp of 20/80, because after trying several adjustments of parameters, there was not enough materials left.

4.2.4.2. Semi-Industrial Scale

The process was scaled up to a semi-industrial scale. While laboratory scale studies investigated the effects of the treatment on the materials and the processability of the materials, semi-industrial scale-up investigated the possibility to manufacture homogeneous yarns and fabrics in larger quantities. The mechanical properties of the manufactured yarns were measured and knitting tests carried out.

At a semi-industrial scale, only medium-length retted hemp (SAMPLE C) and mid-retted linseed flax (SAMPLE D) were investigated. In order to process a larger amount of material, a reactor illustrated in Figure 4.26 was used, treating at least 5 kg of raw material.



Figure 4.26: Above view of the reactor with fibres inside the pierced basket.

The treatments were carried out with the appropriate recipe from laboratory scale, without any changes. The parameters are presented in Table 4.7.

Table 4.7: Treatment conditions at semi-industrial scale.

Alkaline treatment	
Ratio (w/V)	1:20
Time	2 hours
Temperature	80 °C
NaOH	0.2 mol/L
Surfactant	1.5 g/L

Bleaching treatment	
Ratio (w/V)	1:20
Time	30 minutes
Temperature	60 °C
H_2O_2 (50% w/V)	3 mL/L
NaOH (50% w/V)	1 mL/L
Surfactant	1.5 g/L
Activator	2 mL/L

The effect of scaling-up of the alkaline treatment on fibres were studied in previous work carried out by the research group of INTEXTER. The acid soluble lignin content at laboratory scale and semi-industrial scale were compared and only a small variation was probably obtained due to size effects. In this work, the size effects of the treatment on the fibre composition were not studied and the effect of the treatment was considered similar at both scale. This work is considered as a first attempt for producing yarn and fabric. Larger machines were used to cut, draw and produce slivers as described in section 4.2.3.3, and another process of drying was necessary; the wet material was spread out as widely as possible at ambient temperature for a few days in spacious or ventilated conditions. The frame and the slivers formations are presented in Figure 4.27.



Figure 4.27: Strand formation from the revolving flat card (Platts, Werneth, Oldham, England)(left) and a draw frame machine (Platts, Werneth, Oldham, England) (rigth).

Treated fibres were blended with Lyocell to form a sliver in the two different proportions with 50% and 20% of Lyocell. Four slivers were produced:

- Lyocell/hemp: 50/50 (w/w).
- Lyocell/linseed flax: 50/50 (w/w)
- Lyocell/hemp: 20/80 (w/w)
- Lyocell/linseed flax: 20/80 (w/w)

Lyocell was used instead of cotton to calculate the actual amount of each material in the final yarn. Later, the international abbreviation CLY, HA and LI are used to refer to Lyocell, hemp and flax, respectively.

Parameters of the rotor open-end spinning machine were adjusted to obtain yarns of 50 tex with samples containing a blend of Lyocell/hemp in proportion of 50/50 and Lyocell/flax of 50/50. Adjustments were also done to obtain yarns of 80 tex with the blends Lyocell/hemp or Lyocell/flax in proportion 20/80. The following spinning parameters were used:

- Outer rotor velocity: 35,000 min^{-1}
- Coefficient of torsion: 170
- Feed velocity: [0.58-1.84] m/s, according to yarn count and composition
- Production velocity: [46-58] m/s according to yarn count

The evenness of yarns was measured according to the methods detailed in the section 4.1.2.3 of this Chapter. The results of the 50 tex yarns are gathered in Table 4.8.

Table 4.8: Evenness properties of the spun yarns at semi-industrial scale. CLY: Lyocell; HA: hemp; LI: flax.

Yarns	Count (tex)	Irregularity (CV%)	Thins (-50%/km)	Thicks (+50%/km)	Neps (+ 280%/km)
CLY/HA 50/50	50	22.95	199.52	79.81	638.47
CLY/LI 50/50	50	21.01	39.90	39.90	678.37
CLY 100	50	14.04	0.00	239.43	0.00

The introduction of flax and hemp creates irregularity into the yarns. The irregularity is more important for hemp blended yarns than flax blended yarns.

The mechanical properties of the spun yarns were measured, and values are summarised in the Table 4.9.

Table 4.9: Tensile properties of the spun yarns from semi-industrial scale. CLY: Lyocell; HA: hemp; LI: flax.

Material	Count (tex)	Tenacity (cN/tex)	Force at break (cN)	Elongation at break (%)
CLY/HA 50/50	50	9.82 ± 137	490 ± 68	5.16 ± 0.55
CLY/LI 50/50	50	9.53 ± 1.21	475 ± 61	5.86 ± 0.56
CLY/HA 20/80	80	6.50 ± 1.39	514 ± 111	2.93 ± 0.56
CLY/LI 20/80	80	7.95 ± 1.42	634 ± 114	4.56 ± 0.59

It appears that with a high percentage of flax or hemp, the yarns become less deformable but stiffer. It is also revealed that yarns with a high percentage of linseed flax are stronger than yarns with a high percentage of hemp. Moreover, values of tenacity of the samples are closed to 100% cotton yarns (6.8 cN/tex, from (*Mijas, 2022*)), which is the wide employed materials in knitting. The mechanical properties of the yarn were sufficient to be knitted, although the strength requirements for weft knitted fabrics are much lower than the one for woven fabrics. The knitting parameters are summarised in the Table 4.10.

Table 4.10: Knitting conditions and knitted fabric properties. CLY: Lyocell; HA: hemp; LI: flax.

Material	CLY/HA 50/50	CLY/LI 50/50	CLY/HA 80/20	CLY/LI 80/20
Feed rate (mm/turn)	440	455	539	522
Length yarn absorption (mm/stitch)	6.8	7.0	8.3	8.0
Tightness factor	1.0	1.0	1.1	1.1
Stitch/10cm	79 ± 4	74 ± 6	75 ± 1	70 ± 1

By tactile observations of the fabrics, it was noticed that flax fabrics were smoother than the hemp ones for both proportions and fabrics with less Lyocell were rougher. Moreover, the flax fabrics were more deformable than the hemp ones. Those results are in adequation with the mechanical properties, revealing that yarns containing high content of linseed flax had a deformation at break higher than the hemp yarns. The relation between the yarn and the fabrics properties are clearly highlighted: high Lyocell content yarns have high value of deformation at break and in the same way fabrics with high quantity of Lyocell are more deformable than the other ones. The incorporation of hemp stiffens the knitted fabrics.

By visual and microscopic observations, some small shives were detected in the final flax product whereas no hurds were present in the hemp product (Figure 4.28). The presence of shives or hurds, the woody parts of flax or hemp stems, reduces the quality of the fabrics as it affects the sensorial quality, decrease the homogeneity of the fabrics and can lead to future holes.

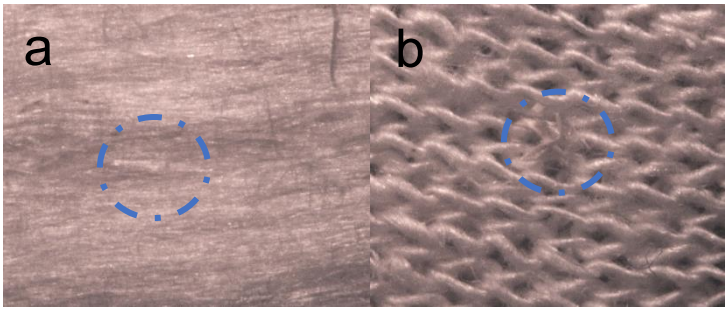


Figure 4.28: Microscopic observations of Lyocell/flaxl-50/50 sliver (a) and fabric (b) with a remaining shive.

Flax shives are generally thinner than hemp ones and could have passed the different step of the process. For further attempt, a stronger mechanical treatment, such as increasing carding passage, can be suggested to remove the impurities.

Moreover, linseed flax slivers and fabrics are whiter than those from hemp, as observed on Figure 4.29.



Figure 4.29: Images of slivers, yarns and knitted fabrics of Lyocell/hemp blends and Lyocell/flax blends. CLY: Lyocell; HA: hemp; LI: flax.

Throughout the process, fibres are lost, and the composition of the blend is modified. The losses mostly originated from comber roll part, when the fibres are combs by an opening roller covered with pins, as observed in Figure 4.30.

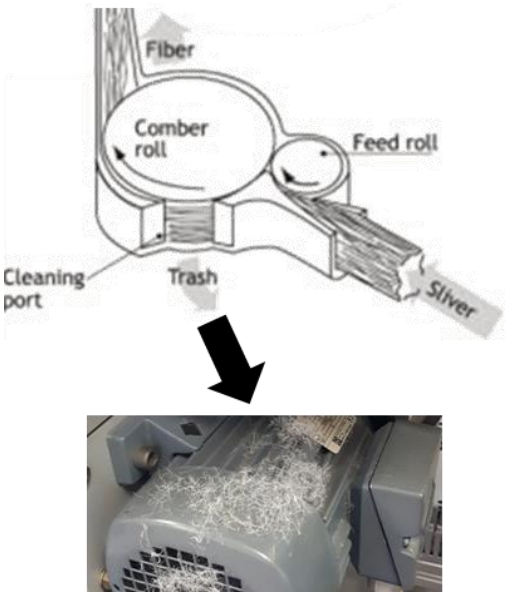


Figure 4.30: Trash coming out from the comber roll of rotor spinning.

The trash was observed by tactile and microscopic analysis (Figure 4.31), and they were mainly composed of non-Lyocell fibres. The hemp and flax fibres might be heavier, stiffer and in larger length heterogeneity than Lyocell, which might hamper their incorporation into the web created by the roller. Hence, the proportion of hemp or flax and Lyocell materials in the final yarn may change compared to the prepared one during carding.

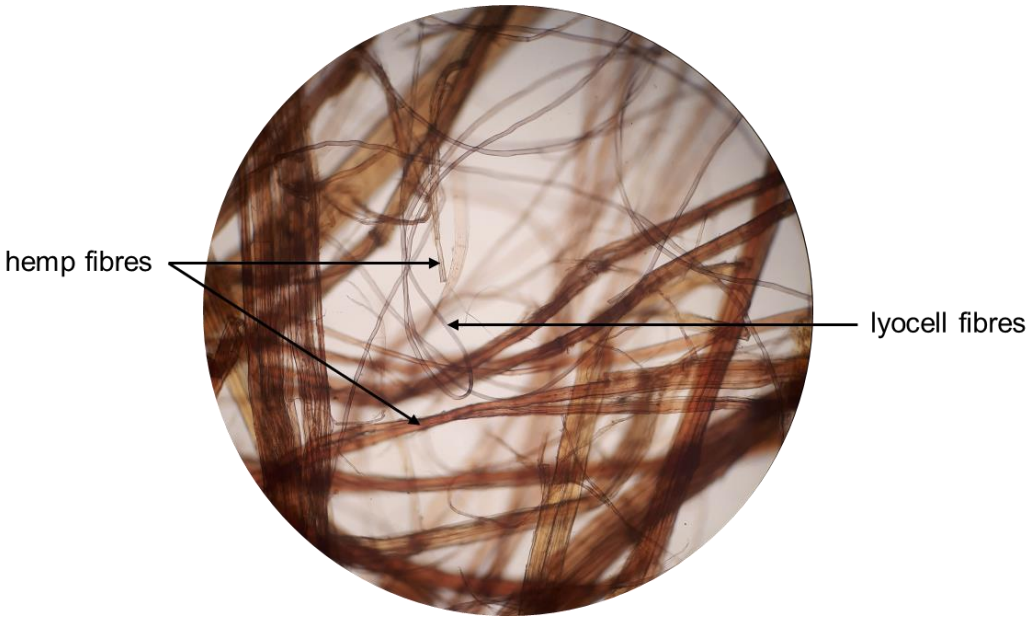


Figure 4.31: Microscopic observation of the spinning trash of Lyocell/Hemp blends slivers.

The final composition of the yarn was measured using the European standard ISO 1833-22:2022 standard. The results are presented in Table 4.11.

Table 4.11: Initial and final composition of yarns.

Blend	Initial composition (%) (w/w)	Final bast fibres content (%) (w/w)	Relative difference in bast fibres content (%)	Final composition (%) (w/w)
CLY/HA	50/50	37 ± 7.8	25.7	63/37
CLY/LI	50/50	48 ± 6.9	3.9	52/48
CLY/HA	20/80	68 ± 5.2	15.1	32/68
CLY/LI	20/80	78 ± 1.6	2.9	22/78

It can be noted that the content of hemp or linseed flax decreased between the initial and the final step of the process. Finally, the highest content of flax or hemp in the final yarn reach (78 ± 1.6)% and (68 ± 5.2)%, respectively. This decrease in bast fibre content is more intense for hemp than for flax. By comparing the experimental count to the nominal count during lab-scale experiments, it was also observed that losses were more important for hemp than for flax. The relative difference between the initial bast fibres content and the measured final one in hemp content is 26% with the initial 50/50 composition and 15% with the initial 20/80 composition and are much more important than the relative differences of sample containing linseed flax. It must be noticed that the standard deviations for the three first samples of the Table 4.11, are over 5% and results might be interpreted carefully. During New analysis would be interesting for more representativeness.

These results show consequent advancement in blended yarns production as in literature only yarns with a lower percentage of by-products fibres were found. In literature, some yarns with hemp hackling wastes and cotton were spun. Rotor-spun yarns of 40 tex with a blend of 30% of hemp and 70% of cotton were produced and higher percentage of hemp in the blends was not reached *(Kozłowski et al., 2013)*. On another hand, 40 tex yarns of 30% cotton and 70% mechanically cottonised textile flax hackling wastes were spun by rotor-spinning *(Cierpucha et al., 2006, 2004)*, but there are no available scientific publications with the rotor spinning of linseed flax waste.

4.3. Conclusions

In this Chapter, the monitoring of materials from the fibres to the yarn was studied. It was demonstrated that hemp grown and harvested in Normandy can be transformed into suitable roves and spun into fine yarns. Yarns with textile range properties were manufactured. Bleached yarns of 24 Nm were spun at laboratory scale using traditional textile flax machine and following a bleaching treatment requiring low temperature and weak alkaline concentrations. With the aim of optimising the textile hemp value chain from the field to the yarn without any addition of chemical products, a yarn having undergone only mechanical treatments was spun to 17 Nm. This work strengthens that the development of a textile dedicated hemp value chain is close to be industrial. Moreover, preliminary trials at industrial scale were done and highlighted the fact that bleached yarns of Nm 18 can be industrially spun and are useable for textile designer. The prices of the European hemp production are high compared to the Chinese production and further improvements are needed to be competitive with the Chinese textile market. In the future, a complete study of the energy consumption should be carried out to measure the environmental impact of the textile chain.

Those recent years, products made from cotton-blended yarns with high flax and hemp content have also become more popular in the clothing market. Waste products are worth

being valorised in blended textile grade yarns. Linseed flax is generally only grown for its seeds and the fibres are low valorised while hemp fibres that are non-aligned and shorter in length than the valuable textile grade hemp fibres also are generally used for low value applications. In this chapter, it was shown that those fibres, with a good retting state, can be processed with a mechanical treatment and an alkaline and bleaching treatment with low temperature to form blended open-end yarns. Depending on the concentration, the alkaline treatment affects both the removal of lignin and the degradation of the cellulose. The tested treatment removed 24% to 50% of lignin content and causes a weak degradation of cellulose. The degree of polymerisation of the material reached values between 1,430 and 1,910 depending on the raw materials, which are evaluated high enough for rotor spinning and knitting applications. 40 tex to 60 tex yarns were formed with blend of cotton in proportion cotton/fibres: 60/40 and 50/50. Yarns of 60 tex can be formed containing 80% of hemp at laboratory-scale. When subjected to the same treatment and same blends, linseed flax slivers are whiter than those from hemp. Tenacity of Lyocell/hemp yarns in proportion 20/80 and 50/50, revealed values of (6.5 ± 1.4) cN/tex and (9.8 ± 1.4) cN/tex respectively. Tenacity of Lyocell/flax yarns in proportion 20/80 and 50/50 revealed values of (8.0 ± 1.4) cN/tex and (9.5 ± 1.2) cN/tex respectively. Those values are closed to 100% cotton yarns (6.8 cN/tex) and suggest sufficient mechanical properties for knitting. Strength at break of Lyocell/hemp yarns in proportion 20/80 and 50/50 revealed values of (517 ± 111) cN and (490 ± 68) cN respectively. Strength at break of Lyocell/flax yarns in proportion 20/80 and 50/50 revealed values of (872 ± 238) cN and (838 ± 158) cN respectively. Yarns with high percentage of linseed flax are stronger than yarns with high percentage of hemp. Rotor-spinning hemp yarns made with 20% of Lyocell and 80% of hemp at 80 tex (12,5 Nm), although thicker, are weaker than the treated and untreated hemp yarns made with laboratory ring-spinning. However, the rotor spinning yarns have an elongation at break (2.93 ± 0.56)% higher than the ring spinning yarns. They are more adapted for knitting than for weaving. Finally, knitted fabrics can be made with 68% of hemp or 78% linseed flax.

For further works, it can be suggested to investigate other proportion of blends and increase the proportion of hemp of flax into the blends. The effect of the treatment within the reactor could be extensively investigated to determine whether all fibres are treated in the same way regardless of their location. A sizing, like an emulsion, could be use during the process at semi-industrial scale to improve the quality of the slivers and limit the friction and therefore the generation of wastes. The evenness of the slivers and the yarns are worth investigating as it reflects the quality of the yarns. To optimise green chemistry in the process, bio-sourced surfactants such as lauryl alcohol could be used, and to reduce the alkaline impact of the treatment, ionic liquid could be used. Survey about the tactile feeling and appearance of the fabrics could be done with a large panel or a panel of experts. Pilling test on fabrics would give more information about the quality of the fabric. Finally, weaving of yarns could be tried.

General Conclusion and Perspectives

The interest of the hemp fibres is growing with the rising societal awareness of environmental issues and the increasing demand for local products. The hemp plants can ensure sustainable solutions for a circular economy and answer the demand for bio-based products. The lack of suitable harvesting and fibre extraction/transformation machinery slowed down the expansion of the hemp fibres at the start of the 21st century. Today, new investments enable the rapid deployment of an industrial value chain. The development of new specific hemp varieties adapted to the needs and areas, and the creation of new machines dedicated to hemp contributes to this great advance.

This thesis aims at participating to the ecological transition and industrial development in France by supporting the deployment of the French hemp textile value chain. In this context, the area of Normandy is deeply studied. The work aims at improving knowledge on the development of hemp fibres within the plant to optimise varietal selection in relation to their morphological and mechanical properties. The thesis work supports industrialisation and investigate the transformation chain and its impact on fibre properties from the fibre extraction to the spinning.

Chapter 1 gives an overview of the general knowledge on hemp fibres. It introduces key concepts and vocabulary for the following chapters. First of all, the description and the history of the hemp plant draw attention to the lack of adapted industrial machinery needed for the extension of textile hemp industry. The second part offers reflections on the development and characteristics of hemp fibres. The structure of the bast fibres, their development and the effect of agronomic practices are reviewed. This highlighted that the genotype and the time of harvesting are possible experimental approaches toward the improvement of fibre qualities. Finally, a state of the art of the possible traditional routes for hemp spinning is given.

Due to improvements in genetics during the last decades, new cultivars are under test and new varieties produced with more adapted characteristics to local territories specificities. The varietal selection performed over many decades aimed at increasing seeds yields or increasing biomass yields for developing dual purposes cultivars without strong analysis on the fibre's quality. Chapter 2 proposes an insight in hemp selection, based on experimental data sets and literature. It establishes the possibility to increase the fibre quality (fineness, tenacity) while assuring a high fibre yield. The breeder's work is an essential expertise for developing the industrial use of hemp fibres for specific applications. Chapter 2 showed that growing late-flowering varieties combining with an early harvest is favourable to obtain fibres with high yields and high quality for textile applications. It opens a new field of industrial developments using varieties which were not used until now in territories, like Normandy, that require an early harvesting to bring in the retted hemp bales in optimal drying conditions. The dioecy of hemp was also studied, corresponding to the natural form of hemp plants. Its superiority in terms of fibre quality for textile applications was reported in the literature when the male and female plants were harvested separately. Chapter 2 detailed little variations in the characteristics of male and female fibres: male plants have a higher fibre content after scutching and hackling while female plants have a higher content in secondary fibres along plant growth and higher mechanical properties at seed maturity.

The first bottle neck for the hemp textile value chain is the lack of mechanisation limiting the production of raw materials. The hemp fibre extraction based on traditional textile flax extraction is considered the most conducive for providing good quality long fibres. The flax process system consists in extracting the fibres from retted stems by scutching and transform them into yarns by combing and wet spinning, citing only the broad outlines of the process. Although the flax process system is considered as a fundamental well-established technology

ensuring the best quality of yarn from long fibres, the hemp system is facing many challenges. Hemp plants are different from flax and parameters must be adapted. The lack of knowledge, appropriate structure and machines prevent farmers from producing hemp. Chapter 3 proposes the study of the production of textile long hemp fibres in the area of Normandy. The development of new harvesting machinery is followed and the adaptation of hemp to the flax extraction process is studied. In the light of the successful results of the last couple of years, seven harvesting machines from Hyler company are now available. An economical approach from the farmers results in high income for farmers that are at the top of their cultivation rotation. The creation of a flourishing new source of high added value fibres is on its way in France with the objective to be at the origin of a renewal of textile industry in Europe.

During scutching a large part of the fibres fall into tows, and the same happens during hackling and should be valorised. These fibres are considered to be of good quality, but they are not aligned and are very variable in length, making them unsuitable for traditional wet spinning. They can be spun by dry spinning to do yarn with high linear density or can be cottonised and spun using an open-end rotor spinning system. In this last case, the cottonised hemp fibres are blended with other materials such as cotton. In the Chapter 4, yarns with low linear density and high percentage of hemp fibres (up to 70%) were produced. The responses of hemp fibres to the cottonisation process were studied. Yarns with different percentages of hemp fibres (50% to 80%) were used to form knitted fabrics. Oilseed flax fibres, which most of the time are little valued and sometimes burned, were also analysed, spun and knitted. Fabrics comprising more than 75% by-product material are formed with yarns reaching 80 tex.

This work demonstrated the feasibility of industrial hemp production in Normandy. The hemp varieties studied in this thesis have shown great capacity for fibres production, but they are not fully optimised for textile purpose yet. Much work on variety selection is still necessary. A reproducible variety, rich in high-quality bast fibres and adapted to climatic conditions and local constraints would improve the competitiveness of the hemp value chain. Combination between the most promising varieties will guide the path towards more and more suitable varieties. Advances in near future can focus on characteristics present in the plants of the variety V4 or V5, for example.

When optimising varieties, one aim is to improve the amount of bast fibre, but as the amount of bast fibre is increased, the secondary fibre content tends to increase as well. These fibres are considered undesirable in the future steps of yarn preparation and wet spinning, but their presence in long fibres and their possible behaviour during scutching and hackling has not been demonstrated. It is questionable whether the secondary fibres stick to the long extracted fibres or fall into the tows or dust. A more detailed study of the tows and long fibres with quantification of primary and secondary fibres would give more information on their behaviour. It would provide important information for variety selection by knowing whether their presence in plants is a real obstacle to variety development.

During varieties optimisation, the filling rate of fibres is also observed. The fibres are maturing by thickening their cell wall and post-synthetic modifications. It is reviewed that the cellulose content increases and protein and pectins are involved in fibre development process. While the plants are growing, a lignification is also observed in the global fibre content. However, this lignification process is not fully described. It is questionable if higher filled fibres necessarily mean higher lignification, or it is independent phenomenon or whether the lignification is occurring with the increasing number of secondary fibres. Spectrometry analysis

such as Raman spectrometry studies on hemp-cross section might be a possible tool for observing this phenomenon.

To complete the high demand in local and vegetal textile fibres, the production of hemp is expected to largely increase in the coming years. Efficient harvesting machines have been developed over the last two years. They have enabled a huge advance in hemp cultivation, although they still require adjustments. More than 2000 hectares of textile hemp are planned to be cultivated in the flax cultivation area from Bayeux to Dunkirk next year. Two more Hyler machines are under construction and two scutching lines were just opened this year for the needs of the “Cooperative Linière du Nord de Caen” as an exponential growth of the hemp surface is targeted. It would be advisable to explore textile hemp cultivation in additional locations with climates that appear conducive to retting. Then, new scutching lines had to be developed near the cultivated area.

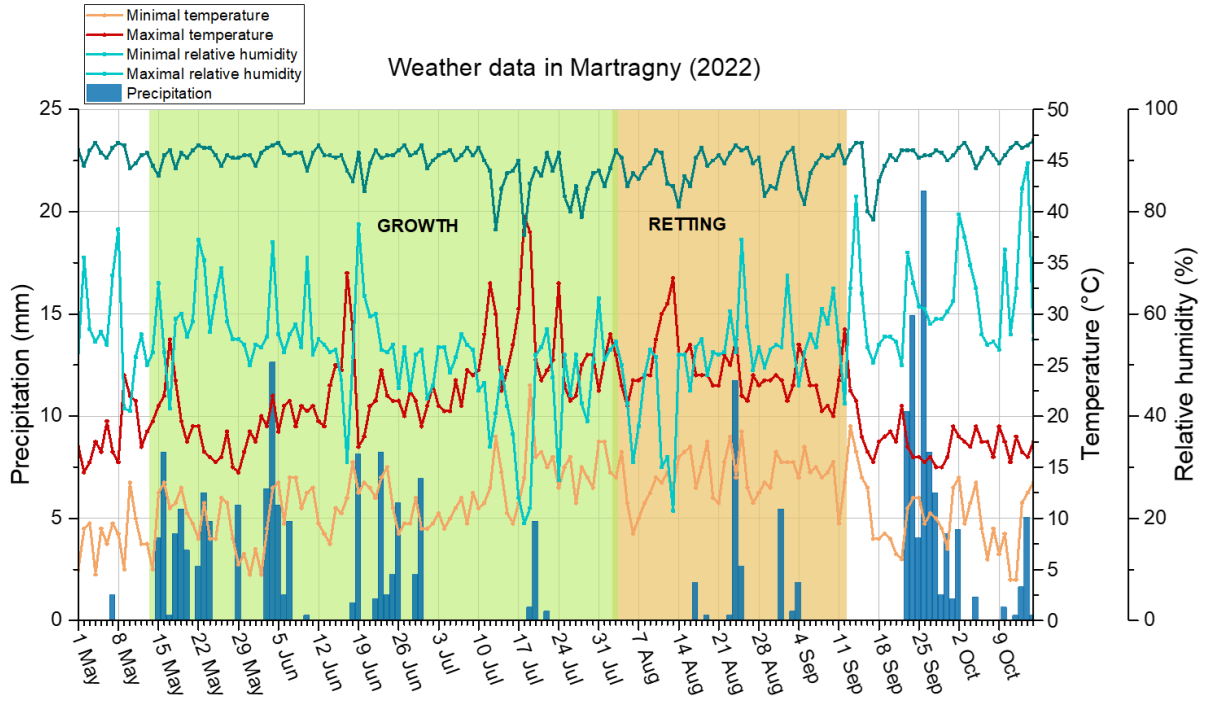
Another solution to meet the strong demand for textile flax and the difficulty to increase the textile flax cultivable area would consist in blending flax and hemp fibres. A blended yarn with properties close to those of a 100% flax yarn is expected. The blend could be done during ribbon formation and according to the chemical and morphological similarity between the two fibres, the ribbon is expected to have good fibres interaction and cohesion.

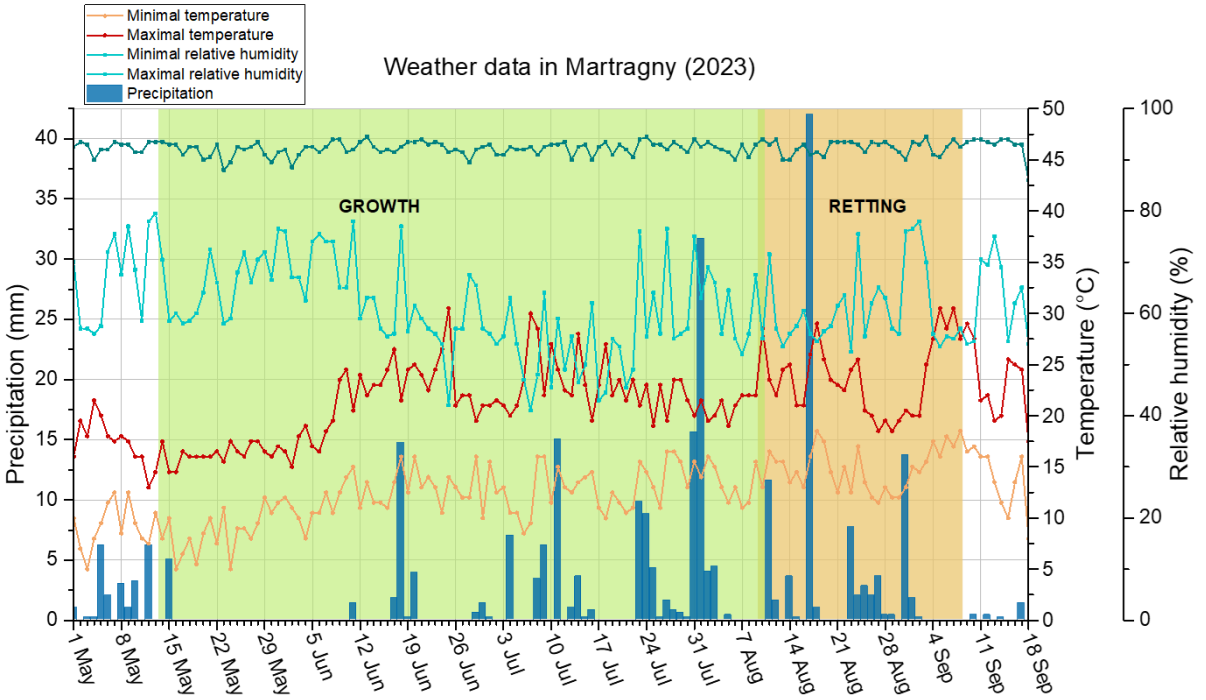
The spinning of linseed flax and disordered hemp fibres led to interesting prospect for the production of blended yarns for textile application. Then the development of the spinning process at larger scale would be advisable. The mechanical and chemical preparations are crucial steps for obtaining quality yarns and those steps might be optimised. For example, green chemistry process could be further investigated.

Finally, a life cycle analysis would provide important information on the environmental impact of the hemp textile value chain in its globally and its interest in comparison to synthetic, cotton or even flax yarn production.

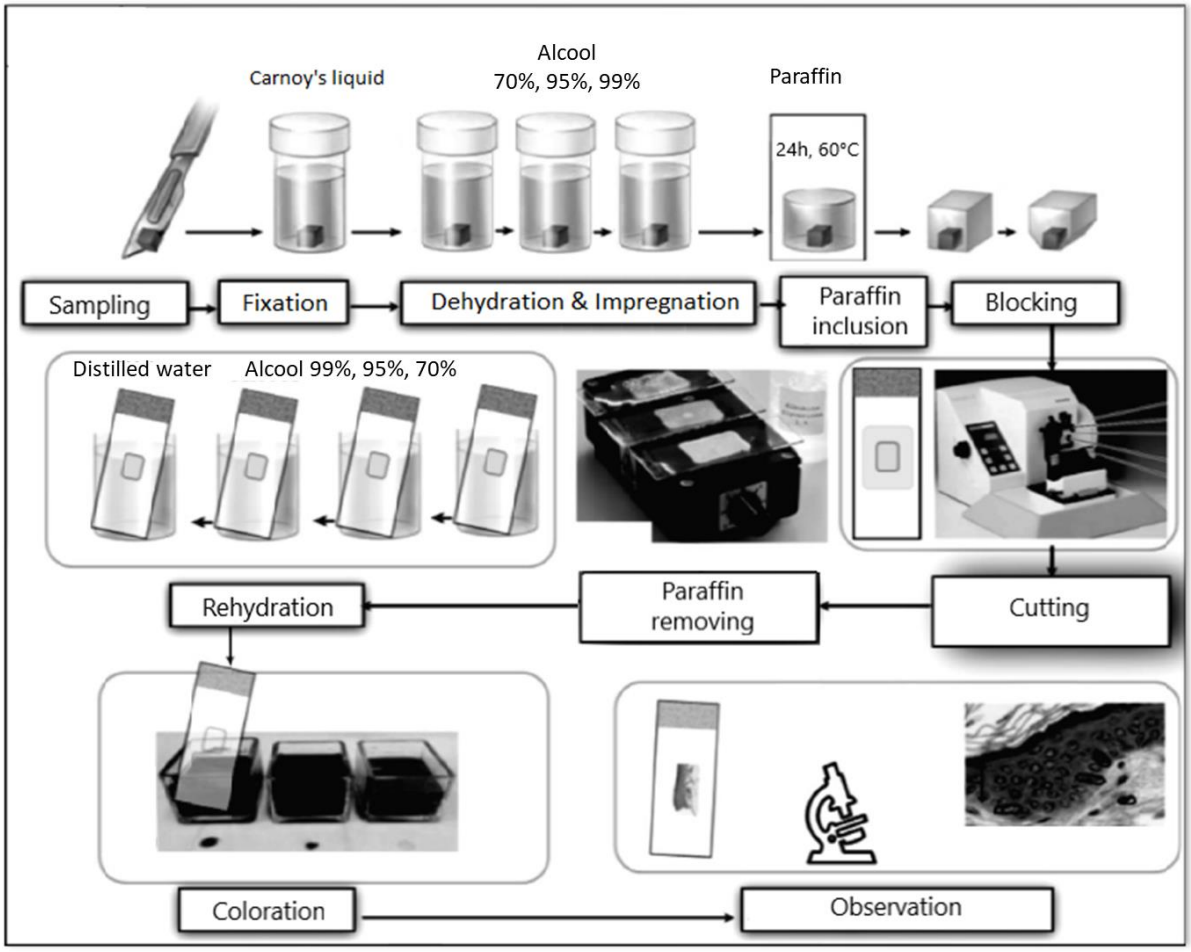
Annexes

Annex 1: Weather data in the field trial in Martragny during hemp growth cycle in 2022 and 2023





Annex 2: Flowchart of histological preparation



Annex 3: Statistical analysis

Statistical tests called Student's test are implemented to determine whether two unpaired batches a and b have significant differences. If the variances of the two groups are identical, it is first necessary to calculate Student's t value:

$$t = \frac{m_a - m_b}{\sqrt{\frac{S^2}{n_a} + \frac{S^2}{n_b}}}$$

With the variance S common to both groups:

$$S^2 = \frac{\sum(x - m_a)^2 + \sum(x - m_b)^2}{n_a + n_b - 2}$$

And m_i is the mean of batch i and n_i is the size of batch i.

The Student's t value and the degree of freedom ddl (calculated according to $ddl = n_a + n_b - 2$) make it possible to identify in the Student's Table the critical value $p\ value$ corresponding to a risk α of 5%. If $p\ value < 0.05$ it can then be considered that the differences observed between the means of the two batches are significant. On the contrary, if $p\ value > 0.05$ the observed differences are not significant. In case the samples have different variances, it is necessary to apply a Welch test. This is an adaptation of the Student test whose Student t values and degree of freedom are calculated with the following formulas:

$$t = \frac{m_a - m_b}{\sqrt{\frac{S_a^2}{n_a} + \frac{S_b^2}{n_b}}}$$

$$dll = \frac{\left(\frac{S^2}{n_a} + \frac{S^2}{n_b}\right)^2}{\left(\frac{S_a^4}{n_a^2 \times (n_a - 1)} + \frac{S_b^4}{n_b^2 \times (n_b - 1)}\right)}$$

The determination of $p\ value$ is carried out by applying the same method as above.

On the graphical representations illustrated in this thesis, when the means of the two batches are not significant, the same letter is indicated; when the means of the two batches are significant two different letters are indicated.

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Titre - Immersion au sein de la chaîne de valeur du chanvre textile : du choix de la variété jusqu'au textile

Résumé : La forte demande actuelle de textile écoresponsable et local justifie le potentiel de la culture du chanvre en France, et ainsi le développement de variétés plus riches en fibres et offrant une meilleure qualité. La culture du chanvre textile, comme celle du lin, est privilégiée dans le bassin s'étendant de Bayeux à Dunkerque où le climat est propice au rouissage au champ. Ce territoire sollicite néanmoins une fauche précoce afin de rentrer les balles de chanvre roui dans des conditions optimales de séchage. La récolte se faisant généralement à la pleine floraison des plantes, la photo-périodicité du chanvre est habituellement un trait majeur de sélection variétale. Cependant, ce travail a montré que les fibres les plus intéressantes d'un point de vue de l'optimisation rendement, et adaptation pour les procédés textile avaient été obtenues à partir de variétés monoïques et dioïques tardives récoltées bien avant la période de floraison, et cela ouvre donc la possibilité d'essais et de développements industriels en utilisant ces variétés jusqu'alors pas utilisées en Normandie. Ce travail de thèse met en avant la possibilité d'obtenir des fibres de chanvre de haute qualité textile en utilisant des procédés d'extraction de teillage/peignage à partir de nouvelles variétés de chanvre monoïques et dioïques. Des essais de chanvre en plein champ visent à collecter des données pour fournir au semencier des éléments en vue de l'amélioration de variété de chanvre, afin de développer des cultivars et de proposer de nouvelles variétés dédiées au textile et adaptées au terroir spécifique.

L'évolution morphologiques des parois cellulaires des fibres de chanvre au cours de la croissance de la plante est explorée et vise à différencier les variétés proposées dans le cadre du projet collaboratif financé par l'Agence de l'Eau Seine-Normandie. De plus, des connaissances sur le processus de développement des fibres de chanvre sont apportées. Des essais ont été réalisés pour optimiser les étapes de fauchage, d'extraction par teillage peignage, de préparation des fibres afin d'obtenir après filature au mouillé des fils de qualité pour l'habillement. Par ailleurs lors du teillage, près de la moitié des fibres tombent en étoupes, des fibres qui sont plus courtes et non utilisables en filature au mouillé. La valorisation des fibres de chanvre mi-longues et non alignées peut se faire en mélange avec une autre fibre, par un procédé de cotonisation suivi d'une filature dite 'open-end'. Des essais à échelle laboratoire puis semi-industrielle ont permis de fabriquer des tissus à forte teneur en chanvre et ainsi de montrer la possibilité de valoriser l'ensemble de la ressource fibreuse pour des applications de textile vestimentaire.

Title - From cultivars to textile application: insight into the textile hemp value chain

Abstract: The current strong demand for eco-responsible and local textiles explains the interest of hemp cultivation in France, and thus the development of varieties exhibiting higher fibre yields as well as better fibre quality. Textile hemp, like flax, is rather grown in the area from Bayeux to Dunkerque, where the climate is favourable to field retting. This territory nevertheless requires an early harvesting to bring in the retted hemp bales in optimal drying conditions. As the flowering time of the plants is a crucial indicator for harvesting, the photoperiodicity of hemp is usually a major trait of varietal selection. However, this work showed that the best compromise between fibre yield and adaptation of the fibres to the textile requirement was obtained from late flowering hemp varieties harvested well before the flowering period. This major discovery opens a new field of industrial developments using varieties which were not used until now in Normandy. The thesis work highlights the possibility of obtaining high textile quality hemp fibres using scutching/hackling extraction processes from new monoecious and dioecious hemp varieties. Field hemp trials aim at collecting data to provide the breeding company with elements for improving hemp varieties, in order to develop cultivars and propose new varieties to its catalogue dedicated to textiles and adapted to the specific terroir (Normandy in our case).

The morphological evolution of the hemp fibre walls during the growth of the plant was explored and aims at differentiating the varieties developed within the framework of a collaborative project funded by the "Agence de l'Eau de Seine Normandie". Additionally, knowledge about the hemp fibre development process is provided. Tests were carried out to optimize the stages of harvesting, extraction by scutching, hackling, and fibre preparation in order to obtain quality yarns for clothing by the wet spinning process. Furthermore, during scutching, almost half of the fibres fall into tow fibres which are shorter and cannot be used in wet spinning. These mid-length and unaligned hemp fibres can be valorised by a cottonization process followed by open-end rotor spinning, in a blend with other fibres. Laboratory and semi-industrial scale trials led to the production of high hemp content fabrics showing the possibility to fully valorised for garment textile applications the hemp fibrous resource provided by the hemp plant.