



Novel fiber value chains & ecosystem services
from sustainable feedstocks

DELIVERABLE 2.1: REPORT ON QUALITY OF CROP
MATERIALS

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GENERAL DATA

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DISCLAIMER

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**Circular
Bio-based
Europe**
Joint Undertaking



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REPORT ON QUALITY OF CROP MATERIALS

Introduction

This deliverable reports on the quality of crop materials that were collected or harvested for the needs of the **FIBSUN** project. It introduces each type of crop, outlining their location and growing conditions, and some key biological characteristics of the selected plants.

The **FIBSUN** project explores both terrestrial and aquatic sources of biomass, developing processes for key plants such as hemp (*Cannabis sativa* L.), poplar (*Populus* spp.), cardoon (*Cynara cardunculus* L.), common reed (*Phragmites australis*), and cattail (*Typha* spp.). These crops were carefully selected for their ability to mitigate desertification, organic matter loss, and biodiversity decline in a wide range of pedoclimatic conditions, from hot and dry to cold and moist climates.

Analysis of the physical and chemical properties of these species, essential for developing new technologies within the scope of FIBSUN, is also reported in this deliverable. Each type of material had its own measured properties. These properties were chosen according to the characteristics of the end products made from them. (Fig 1.1)

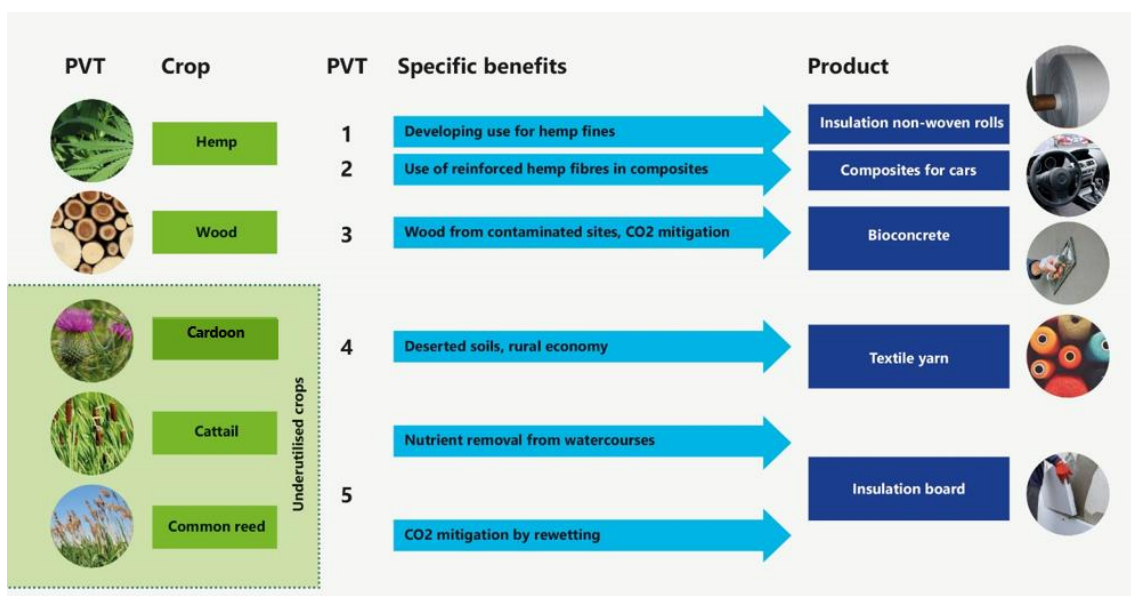


Fig 1.1. Diagram of the different materials used per PVT and the end products for which they have been selected.

The plan for the deliverable will be presented as follows:

For each type of plant, an initial crop site and harvest description will be given, ensuring to know how, where and when the plants were grown.

A second part will be devoted to a description of the plant itself, its physical characteristics and, if necessary, methods for identifying it.

Finally, a third part will be dedicated to the results of physical and chemical analyses of these plants, with a preliminary description of the methods used to conduct them.

Hemp (*Cannabis Sativa* L)

Fibers extracted from these plants will be used to produce rigid insulation boards in PVT1 and non-woven for automotive composite products in PVT2.

Crop site and harvest description

Crop is located in Germany, Waldhegenich in Bühl (fig 1.2), a nature and landscape conservation area. The Waldhägenich nature and landscape conservation area is located on the territory of the town of Bühl and the municipalities of Bühlertal and Ottersweier in the district of Rastatt in Baden-Württemberg.

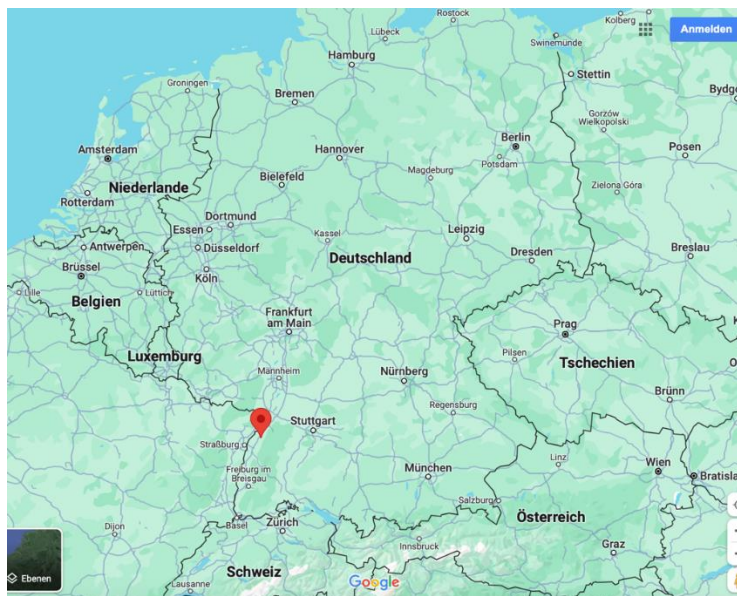


Fig 1.2. Crop location in Germany

In 2024, an organic hemp crop of Finola 2 variety was cultivated on a 2.7-hectare plot of sandy soil that had previously been used for corn and, for the third consecutive time, for hemp. Sandy soil lacks nutrition, rapid soil warming, and dryness. On this type of soil, water permeability is high but holding capacity is low.

Sowing occurred on May 17th, 2024, with a combine harvester (fig 1.3) with modified Kemper corn deep-cut cutting unit (bs = 4.5 m) and special chopping device to shorten the hemp plant stems to approx. 450-600 mm. Central swath storage of the grain-free, shortened whole plant straw for drying and roasting the stems.



Fig 1.3. Combine harvester used to collect crops. Field after harvest.

Plant identification & description

Description of the FINOLA2 oilseed hemp variety (fig 1.4):

- Type: Dioecious, with approximately 50/50 male and female plants
Main uses: Large size hempseed and green material biomass
Flowering: 25 to 30 days after sowing male flowers appear
- FINOLA2 is an auto flowering dioecious hemp variety. The flowering process typically begins between 35 to 40 days after sowing, with male flowers being the easiest to identify at first. FINOLA2 also has a significant large seed, with a thousand seed weight (tsw) of approximately 16-18 g/, and a very low THC content (<0.10%) at maturity. With adequate fertilisation and favourable growing conditions, typical yields of grain range from 1.5 to 2 metric tonnes per hectare in Central Europe. FINOLA2 is suitable for cultivation in Southern Europe, the United States and other regions below 50° N latitude. Grain yields are less than optimal at latitudes above 50 North.



Fig 1.4. Views of the Finola2 hemp variety fields

Plant characteristics:

- Average size whole plant: approx. 200 cm
- Average stem diameter 1 cm
- Average plant maturity / harvest time: approx. 100-130 days after sowing

Plant composition and properties

Methods

In **PVT 1**, super short hemp fines (P1H1.SKF) and short hemp fibers (P1H1.KF) were characterized based on their physical and ultrastructural properties. This included particle size analysis using QICPIC, scanning electron microscopy (SEM) for fiber morphology assessment, chemical composition analysis (lignin, cellulose, hemicellulose content) done using the Van Soest protocol, crystallinity assessment using X-ray diffraction (XRD), thermal properties via thermogravimetric analysis (TGA), and sorption analysis with dynamic vapor sorption (DVS). For **PVT 2**, in addition to these analyses, the tensile properties, structural composition with Fourier-transform infrared (FTIR) spectroscopy, and diameter of the technical hemp fiber (P2H2.VF) were examined.

Results

Morphological characteristics:

Particle size analysis with QICPIC: The particle size (length, width) of the hemp fines (P1H1.SKF) and super short fibers (P1H1.KF) were examined using the dynamic image analysis tool (QICPIC). Approximately seven replicates were analyzed, with fiber counts around 63,430. Fig.1.5a illustrates the length and width distribution, while Fig.1.5b highlights the median and mean size. The analysis revealed that P1H1.KF fibers are roughly 35% longer than P1H1.SKF fibers (p-value = 0.009, one-way ANOVA with a confidence level of 0.05). Although P1H1.KF fibers were 20% wider than P1H1.SKF fibers, this difference was not statistically significant (p-value = 0.06). Technical fibers are not examined with QICPIC because they are long fibers and not suitable for measurement with such an analysis tool.

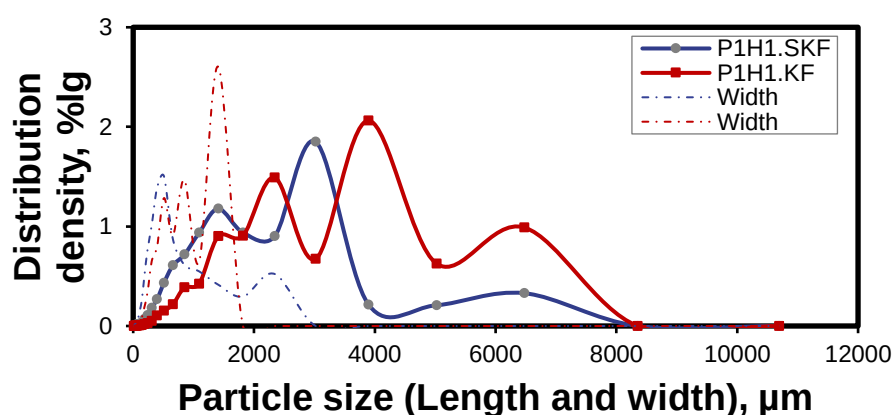


Fig 1.5a. Particle size distribution of P1H1.SKF and P1H1.KF.

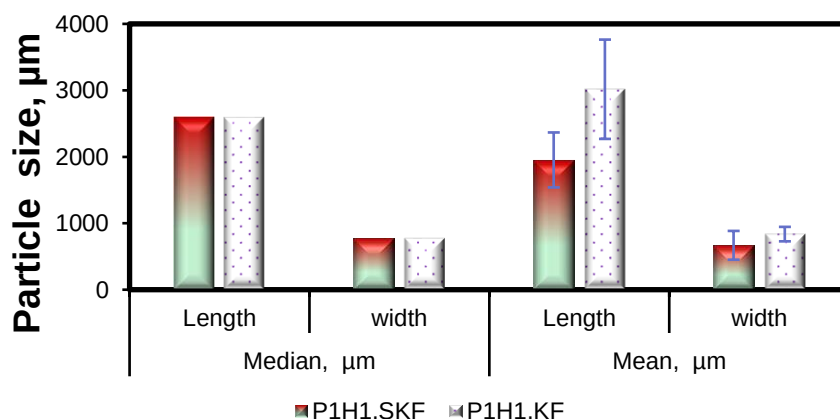


Fig 1.5b. Median and mean of the particle size distribution for P1H1.SKF and P1H1.KF.

Chemical Composition

The chemical composition (Table 1.1a) reveals that hemp fines (P1H1.SKF) exhibit a greater overall lignin content. Lignin content is also more pronounced in P1H1.KF than P2H2.VF (presented in Table 1.1b), but less so than in P1H1.SKF. This outcome is

mainly because lignin is often more concentrated in the core layers of plant stems. The mechanical process of obtaining the technical fibers (P2H2.VF), involves separating the long, fibrous outer layer (rich in cellulose) from the inner woody core (rich in lignin). The fines and shorter fibers contain a higher proportion of these core layers, leading to a higher lignin content.

Table 1.1a. Compositional analysis of super short (P2H2.SKF) & fiber fines (P2H2.KF).

Short Hemp/fines	Soluble content, %	Hemicelluloses, %	Cellulose, %	Lignin, %	Residual ash, %
P1H1.SKF	22.5 ± 3.0	10.9 ± 3.9	51.6 ± 19.1	21.6 ± 18.9	1.9 ± 1.2
P1H1.KF	8.8 ± 1.8	9.7 ± 4.2	69.3 ± 14.1	6 ± 2.8	3.9 ± 1.5

Table 1.1b. Compositional analysis of technical fibers.

Technical Hemp	Soluble content, %	Hemicelluloses, %	Cellulose, %	Lignin, %	Residual ash, %
P2.H2.V	5.0	6.9	70.7	14.3	3.1

TGA and **FTIR** corroborate the findings presented in [Tables 1.1a](#) and [\(b\)](#). A primary distinction lies in the initial mass loss at lower temperatures ([Fig 1.6a](#)). The TGA curve for P1H1.SKF exhibits a more rapid weight decrease, suggesting a higher concentration of thermally labile components (i.e., greater soluble content) compared to P1H1.KF and P2H2.VF. The inorganic content of P1H1.SKF is notably higher ($26 \pm 3\%$) than that of P1H1.KF ($19 \pm 2\%$) and P2.H2.VF (21%). This suggests P1H1.SKF might be a more suitable insulating material. **FTIR** analysis ([Fig 1.6b](#)) reveals a higher soluble content and a more pronounced methylene group peak associated with lignin in P1H1.SKF. No differences are notable in the spectrum of P1H1.KF and P2H2.VF.

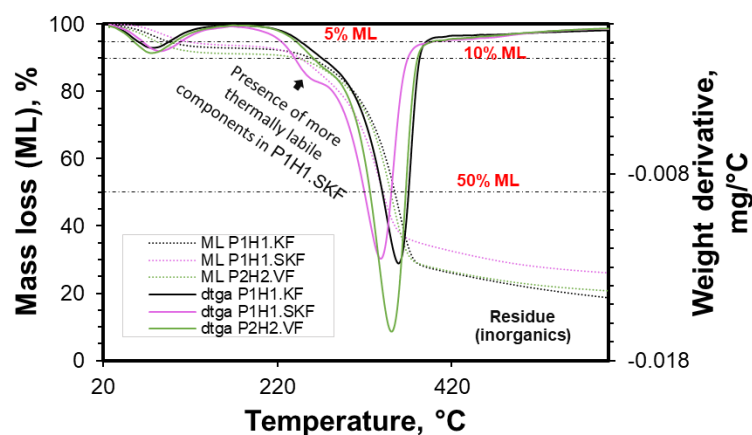


Fig 1.6a. Thermogravimetric analysis of the hemp fractions: short fines (P1H1.SKF), short fibers (P1H1.KF) and technical fibers (P2H2.VF).

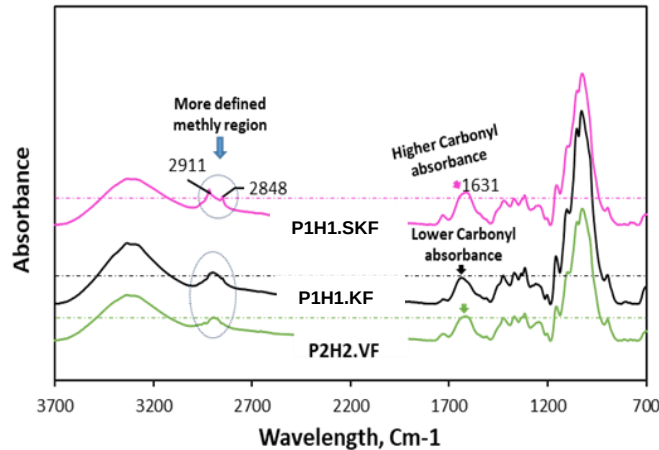


Fig 1.6b. Fourier transform infrared (FTIR) spectrum of the hemp fractions: a) short fines (P1H1.SKF), short fibers (P1H1.KF) and b) technical fibers (P2H2.VF).

XRD analysis did not reveal any significant differences in the crystallinity index (I_c) among the hemp substrates. The I_c values for P1H1.SKF, P1H1.KF, and P2H2.VF were found to be **63.6%**, **64.2%**, and **62.0%**, respectively. However, we note that the high I_c for P1H1.SKF may be due to various factors, including that the processing of the straws into fibers may have resulted in the inadvertently favored formation of crystalline structures in the cellulose. Besides, the high lignin content may have resulted in an interaction with cellulose in ways that promote crystallinity, even in the presence of a lower cellulose content.

Dynamic vapor sorption (DVS)

DVS analysis of the hemp samples is presented with the sorption curve (Fig. 1.7). The sorption curves illustrate the relationship between moisture content (EMC) and relative humidity (RH) for each hemp sample, with the slope indicating sorption rate. Among the three samples, P1H1.SKF exhibits the highest EMC at any given relative humidity (RH), suggesting it can absorb and retain more moisture compared to P1H1.KF and P2H2.VF. The DVS analysis supports the chemical composition findings, highlighting that the very short fines (P1H1.SKF) contain a higher concentration of hydrophilic functional groups.

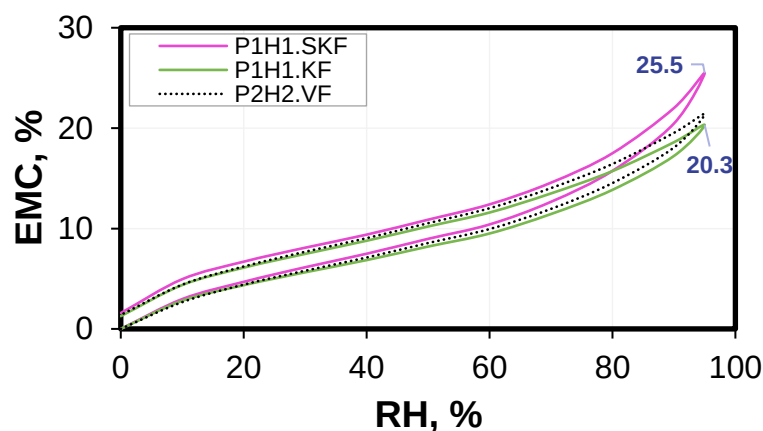


Fig 1.7. Sorption hysteresis of the hemp biomass (super short fines (P2H2.SKF), short fibers (P2H2.KF) and technical fibers (P2H2.VF)).

Mechanical strength

The tensile properties (Fig 1.8) indicate that the tensile strength of **P2H2.VF** is **356.6 ± 198.3 MPa** and the modulus is **32.6 ± 19.7 GPa**. The average diameter of the fiber bundles was **103 ± 47 μm**.

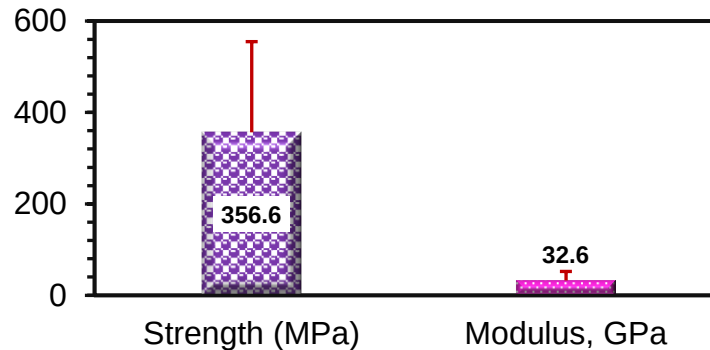


Fig 1.8. Tensile strength and modulus of P2H2.VF.

Conclusions:

- Hemp biomass fractions have been collected and examined.
- Super short fibers (P1H1.SKF) are 30% shorter and 20% lower in width than short fibers (P1H1.KF).
- Chemical and structural component analysis show variations in the properties of the hemp fractions with substantially higher lignin composition of P1.H1.SKF.
- DVS analysis reveals higher overall moisture sorption for the super short hemp fines while XRD did not present substantial differences in crystallinity index.

Recommendations for trials:

The notably short fiber lengths, particularly for P1.H1.SKF (1500 μm to 2500 μm), could hinder the production of thermal insulation panels due to potential limitations in densification and the possibility of increased dust content. Additionally, P1.H1.SKF shows a high soluble content, which negatively impacts sorption characteristics. Such hygroscopic behaviour may cause issues with thermal insulation performance and durability. These observations from the detailed characteristics are indicative with no implications for WP1, as the fines are generated based on BAFA's mechanical throughput (to be discussed in D2.2).

Therefore, planned verification is suggested to ascertain the most optimal material for substitution with the reference wood fiber. This will ascertain the level of impact and feasibility for P1.H1.SKF, despite the fiber properties, to achieve the planned activities as agreed in the grant agreement.

Wood from contaminated soil (Poplar (*Populus Sp.*), Willow (*Salix Sp.*), Black locust (*Robinia Pseudoacacia*))

Crop material concerns trees that have grown on contaminated soils (i.e. soils containing heavy metals, polycyclic aromatic hydrocarbons, polychlorinated biphenyls or volatile organic compounds). Wood from these trees will be used in the mix with cement to produce insulating bricks. An **insulating brick** made from a combination of **cement and biomass** is a sustainable construction material designed to provide thermal insulation while reducing environmental impact. The composition typically includes a blend of **cement**, which provides structural strength, and **biomass materials** such as hemp fibers, wood chips, or agricultural residues. These biomass components serve as lightweight aggregates that enhance the brick's insulating properties by trapping air and reducing thermal conductivity.

Crop site and harvest description

Trees were collected the 6th of February 2024 on a former Industrial site (Becker industrial, a paint and varnish industry, closed in 2011) in urban areas of Le Pont-de-Claix, near Grenoble in France (Fig 2.1). The site is now operated by Crisalid, a cooperative society that brings together major public stakeholders, SMEs and start-ups around projects of soil study, remediation and refunctionalization.

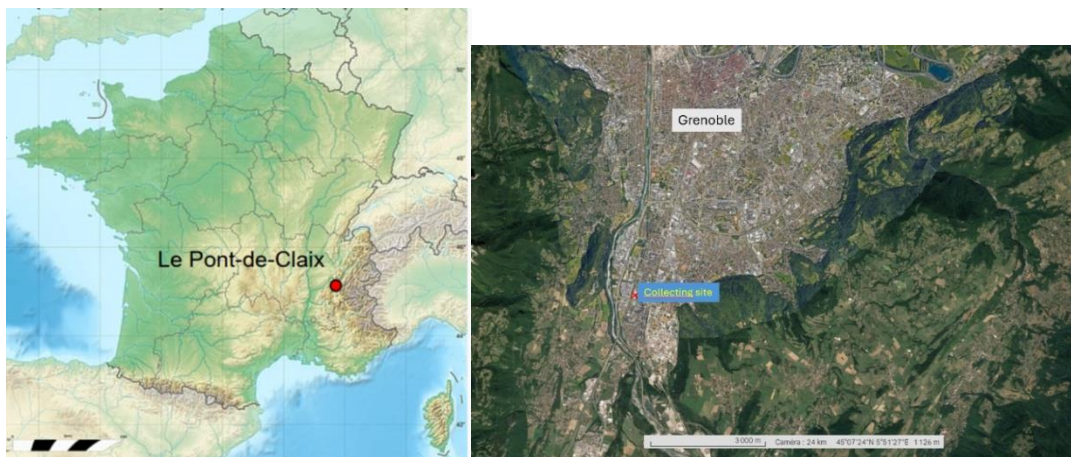


Fig 2.1. Trees collecting site location near Grenoble, France

Four trees (Poplar P1, Black locust A1, Willow S1, Black locust A2) were collected at the locations indicated on the view (Fig 2.2) below by red crosses.



Fig 2.2. Top view of collecting site location and collected tree's location

The collected trees are endemic to the region and have grown naturally on the site since the factory closed.

The trees were collected by lumbering using a chainsaw. The collected material was taken from the trunk and main branches of the tree. Branches smaller than 4cm in diameter were not collected.

Tree fraction:

At the time of collection, the means available on site did not allow for: weighing, crushing or transporting the small branches which were left on site, so the usable/non-usable ratio could not be measured.

At this time (February 6, 2024) hardwoods are also leafless so the ratio of leaves to tree could not be measured either.

Nevertheless, a study by the Quebec Ministry of Forests, Wildlife and Parks on six-year-old Poplar (Note N°7; 1977); revealed that dry biomass was distributed as follows:

- Trunk: 6,929 kg/ha or 76.3% of total weight
- Branches: 1,078 kg/ha or 11.8% of total weight
- Leaves: 1,074 kg/ha or 11.8% of total weight

The usable fraction of the branches would be to determine, but we can estimate the total usable fraction of the collected trees to be up to 80%.

Use of residues:

It is generally recommended to leave a significant portion of slash (branches, leaves, stumps, bark) on the ground to maintain the fertility and health of the forest ecosystem.

The proportion of slash to be left in place after a forest cut varies according to the mineral richness or poverty of the soil, tree species, climate, and forest management objectives.

These recommendations can be found in guides to good forestry practice (in France, these are issued by French Environment and Energy Management Agency (ADEME) or the national forestry office (ONF)). In the case of contaminated soils, however, the question may arise as to whether it is a good idea to leave these slashes in the forest,

since the contaminants could then find their way back into the elementary biodiversity chain that contributed to their degradation. This subject is not covered in this study.

A possibility of slash management practices can also be an off-site export and slash transport for panel production (particleboard, MDF) for branches; Production of biofuels or compost for leaves and branches.

Identification:

The wood species identification as shown in Fig. 2.3a-c is based on the analysis of the cellular organization of wood tissues (cut according to radial, tangential, and transversal orientation). The microscopic characteristics for some species are similar and therefore cannot be microscopically differentiated, in which case the expertise will stop at the determination of the family, the botanical genus, and / or a grouping of species. The main elements observed include the shape and distribution of vessels in hardwoods, as well as the shape of bordered pits. The analyses were conducted by FCBA's Testing and Simulation Laboratory in Bordeaux (France).

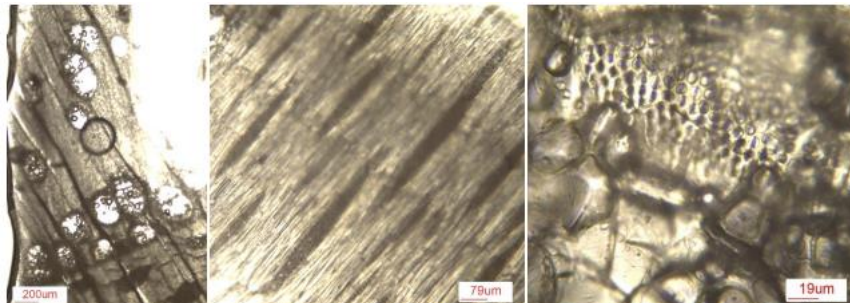


Figure 2.3a. Transversal and tangential cuts for black locust A1 (*Robinia pseudoacacia*) samples

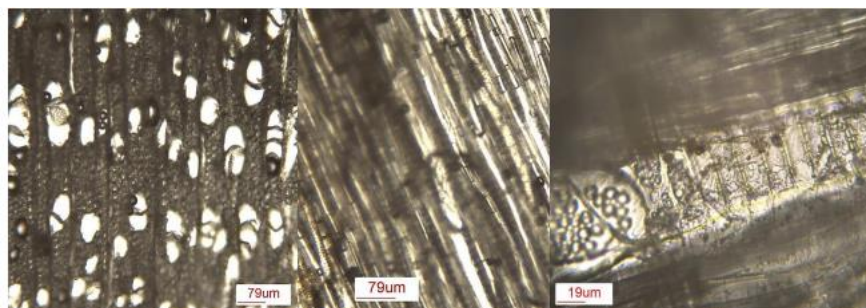


Figure 2.3b. Transversal and tangential cuts for Poplar P1 (*Populus sp.*) samples

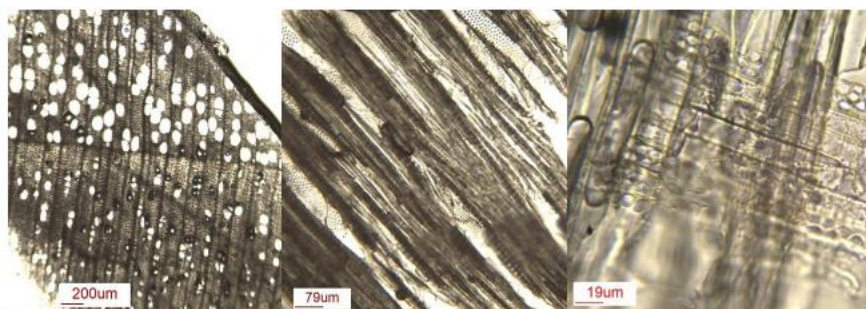


Figure 2.3c. Transversal and tangential cuts for Willow S1 (*Salix sp.*) samples

Tree characteristics:

Based on the number of annual rings in the wood at the base of the tree (fig 2.4), all trees are 9 years old.

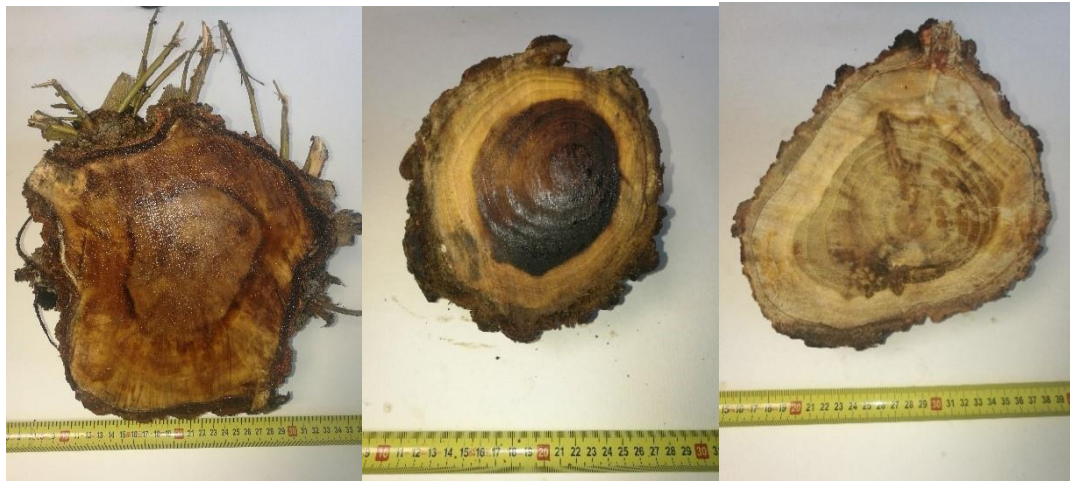


Fig 2.4. From left to right: Base section of Poplar P1, Black locust A1 and Black locust A2

Tree base diameters and heights:

- Poplar P1: 20cm Height: 4m
- Black Locust A1: 13 cm (the tree separated early in two main trunks) Height: 4.3m
- Black Locust A2: 18 to 20 cm Height: 5 m
- Willow S1: 21 cm Height: 4.2 m.

Soil composition

Soil composition was analyzed at every tree's location. Analyzed soil contaminants analyzed were Hydrocarbons, PAHs (polycyclic aromatic hydrocarbons), PCBs (polychlorinated biphenyls) and the main heavy metals present in the soil. The soil analyses were carried out by Envisol, part of the Crysaliid project. The following **Table 2.1** shows the main results from this soil analysis. Four soil samples were collected (Poplar P1, Black locust A1, Willow S1, Black locust A2). A soil sample consists of a composite mixture of at least 5 soil samples homogenized in a bucket. The soil samples were then put in suitable bottles following the analysis. Soil samples were collected with a manual auger.

Table 2.1. Soil composition for the four harvested trees.

Components (in mg/kg of soil dry matter)	Poplar P1	Black locust A1	Black locust A2	Willow S1	Range of values commonly observed in ordinary soils of all grain sizes in France (from ASPITET programme (INRA - Denis Baize, as of 18 August 2010))
Hydrocarbon index C10-C40	200	150	120	<20	-
Sum of PAHs	0.59	0.11	0.61	-/-	-
Sum of 7 PCBs	3.70	18	9.5	0.07	-
Chromium (Cr)	100	86	130	22	10 to 90
Nickel (Ni)	28	28	29	25	2 to 60
Copper (Co)	27	27	46	14	2 to 20
Arsenic (As)	9	7	12	8	1 to 25
Cadmium (Cd)	0.5	0.8	0.9	<0.4	0.05 to 0.45
Mercury (Hg)	0.8	0.2	0.4	<0.1	0.02 to 0.1
Lead (Pb)	360	400	490	20	9 to 50

The presence of hydrocarbons, PAHs, PCBs and relatively high levels of heavy metals compared to ordinary soils values in France shows that the soils from which the trees were extracted have been artificially contaminated by pollutants from the previous or surrounding industrial and urban activities.

Only the soil from Willow shows no pollution from PAHs, HCT, or PCBs and has low concentrations of metal(oid)s compared to the other sampling sites. The highest PAH pollution is found in soils from Black Locust A2 (Fluoranthene) and Poplar P1 (Naphtalene), the highest PCB pollution in Black Locust A1, the highest HCT pollution in Poplar P1, and the highest concentrations of metal(oid)s in Black Locust A2.

Plant composition and properties

Methods

The approach adopted in this PVT is global to be closer to industrial reality. The recoverable part of the tree is treated and analyzed as material of its own. Sections of wood trunks representative of the tree have been chipped, and a portion of the bark has been selected to perform an average, non-localized analysis of the material.

Analyzed samples and prelevement methods:

The analysis was carried out on parts of the tree trunk. Once the tree had been truncated, the selected elements were cut into pieces approximately 60-70 cm long. Of these sections, part belonging to the tree trunk was debarked by hand and the debarked logs were shredded. Around 150 grams of unground extracts (full bark samples and wood chips) were sent for characterization of pH, pH buffer, ash levels, and ICP analysis of contaminants. (see fig. 2.5)

Around 200 g of these chips were ground and sieved to 500 µm. A portion of the bark was also ground and sieved to a mesh size of 500µm. 20 grams of each ground sample were sent for chemical composition analysis.



Fig 2.5. Parts of the samples analyzed (bark and wood) for characterization of pH, pH buffer, ash levels and ICP analysis of contaminants of reference poplar.

For the purposes of the project, commercial poplar and locust logs were ordered by FCBA to carry out milling and optimization tests to avoid wasting the samples taken from contaminated soils. Hereafter, these samples are called “reference samples”, while the ones from contaminated soils are called “contaminated soil samples”. Reference black locust wood was delivered without bark.

Table 2.2 presents the wood biomass examined. The wood samples were analyzed for particle size (particle size analysis), pH, ash content, buffering capacity, vapor sorption by DVS, and ICP measurement for metallic and inorganic elements.

Table 2.2. Description of wood biomass.

Nature	Description	ID	Abbreviation
Vicat reference (Sourced)	Hemp	P3.H18	-
Reference samples (Market samples, sourced from the sawmill)	Poplar wood reference	P3.H5.S12	S12
	Poplar bark reference	P3.H5.S13	S13
	Locust wood reference	P3.H4.S14	S14
Biomass derived in the FIBSUN project (Harvest from contaminated soil): A1 and A2 represent Black locust obtained from two separate contamination sites	Poplar bark P1	P3.H1.S5	S5
	Poplar wood P1 (no bark)	P3.H1.S4	S4
	Willow wood (no bark)	P3.H2.S6	S6
	Willow bark	P3.H2.S7	S7
	Locust wood (A1)	P3.H3.S8	S8
	Locust bark (A1)	P3.H4.S9	S9
	Locust wood (A2)	P3.H4.S10	S10
	Locust bark (A2)	P3.H4.S11	S11

Results

Chemical composition and extractive content

The results (Table 2.3) show comparable chemical compositions between samples S4 and S12 (poplar wood, reference and from contaminated soil) and between S5 and S13 (poplar bark, reference and from contaminated soil). Notably, the fraction of hemicellulose in the wood biomass is high but falls in the range of known values (20-30%) for hardwoods like locust, willow, and poplar (Berglund 2018). Some studies have also reported that there are differences between 15 to 35% in hardwoods (Abik et al. 2023). Such variability is ascribed to factors such as tree age with younger trees characterized by higher hemicellulose contents, growth conditions, and sampling methods. Rapidly growing trees in this case may also exhibit higher hemicellulose content, as this polysaccharide is crucial for cell wall development.

Table 2.3. Chemical composition and extractive content (obtained by acetone solubilization) FIBSUN collected wood biomass (S4, S5, S8, S9, S10, S11, S6; S7), samples from sawmills (S12, S13; S14) and Vicat Reference (VH). The values represent mean with deviations presented in ⁰.

	ID	Cellulose, wt. %	Pectin, wt. %	Hemicellulose, wt. %	ASL, wt. %	AIL, wt. %	lignin (ASL+AIL), wt. %	Extractives, wt. %
Reference poplar wood	S12	39.4 ^(0.7)	2.3 ^(0.0)	27.1 ^(0.3)	2.3 ^(0.0)	19.8 ^(0.1)	22.1 ^(0.1)	2.2 ^(0.0)
Reference poplar bark	S13	21.5 ^(2.1)	7.2 ^(0.3)	20.8 ^(0.4)	1.6 ^(0.1)	28.0 ^(0.0)	29.6 ^(0.1)	3.1 ^(0.1)
Poplar wood (P1)	S4	35.2 ^(2.1)	2.7 ^(0.1)	27.7 ^(1.4)	2.2 ^(0.1)	20.6 ^(0.3)	22.7 ^(0.3)	3.1 ^(0.1)
Poplar bark (P1)	S5	21.2 ^(1.4)	5.9 ^(0.3)	21.1 ^(0.5)	2.2 ^(0.0)	28.1 ^(0.6)	30.3 ^(0.6)	4.0 ^(0.1)
Reference locust wood	S14	34.9 ^(0.2)	2.5 ^(0.1)	25.4 ^(0.4)	1.5 ^(0.0)	26.0 ^(0.8)	27.5 ^(0.8)	5.1 ^(0.4)
Locust wood (A1)	S8	35.6 ^(1.3)	2.5 ^(0.0)	28.3 ^(0.3)	1.9 ^(0.1)	20.4 ^(0.5)	22.2 ^(0.4)	2.5 ^(0.0)
Locust bark (A1)	S9	15.3 ^(0.5)	6.2 ^(0.3)	19.9 ^(0.1)	4.1 ^(0.2)	24.1 ^(1.5)	28.1 ^(1.2)	3.5 ^(0.0)
Locust wood (A2)	S10	34.5 ^(1.3)	3.0 ^(0.0)	28.1 ^(0.3)	2.8 ^(0.0)	21.9 ^(0.1)	24.7 ^(0.2)	2.4 ^(0.1)
Locust bark (A2)	S11	17.9 ^(0.2)	5.8 ^(0.3)	18.4 ^(0.4)	4.6 ^(0.1)	22.8 ^(0.5)	27.4 ^(0.4)	4.2 ^(0.1)
Willow wood (S1)	S6	37.2 ^(2.2)	2.6 ^(0.2)	28.9 ^(1.3)	2.3 ^(0.0)	19.3 ^(0.0)	21.6 ^(0.0)	2.1 ^(0.1)
Willow bark (S1)	S7	18.3 ^(0.5)	5.4 ^(0.3)	25.3 ^(0.9)	2.8 ^(0.2)	27.7 ^(0.1)	30.5 ^(0.2)	12.1 ^(0.8)
Vicat Hemp	VH	33.8 ^(2.5)	0.6 ^(0.0)	23.2 ^(0.1)	1.4 ^(0.0)	21.6 ^(0.1)	22.9 ^(0.4)	1.6 ^(0.0)

The most obvious implications on composition of the biomass were wood relative to bark. The trend was consistent for all samples with cellulose and hemicellulose contents higher in wood than relative bark. And pectin, lignin and extractive contents are higher in bark than relative wood. Comparing poplar samples shows that while lignin and hemicellulose components are similar, extractive content was higher, and cellulose was about 12% lower in the wood from the site (S4) compared to the sample from the sawmill (S12). In the case of barks, the components were similar, except for higher levels of pectins (30%) in S13 than S5. The black locust samples are comparable with each other but have a lignin content of 3 to 5 points lower than that of the reference locust, and a hemicellulose content around 5 points higher.

Aside from the notably higher levels of lignin, pectins and extractives in wood, S4, S8 and S10 (all wood samples from the contaminated site) are wood biomass closely related to VH in composition.

Ash content

Ash content was determined using five replicates of the wood granules (1 g each). The samples were pre-dried at 103 °C for 24 h before incinerating in a furnace at 550 °C for about 4 h. Fig 2.6 shows the ash contents of the wood samples. The reference Poplar

sample without bark did not show any ash content after the incineration. Generally, ash content is rather influenced by bark content. For example, when comparing poplar reference and poplar from contaminated soils, bark samples produced about the same ash contents, which were higher than the batches without bark. However, the substantial ash content in the contaminated sample of the poplar without bark may suggest traces of inorganic contaminants. Similar comparison does not show a meaningful difference in ash content of locust (sawmill and contaminated (FIBSUN related harvest)). Wood with bark and from contaminated soil may interact differently with cement due to their higher ash content compared to the reference Vicat hemp. For instance, the implications of high inorganic or ash content as it concerns wood additives in concrete mixtures can potentially lead to cement hydration, causing a reduction in the compressive and flexural strength of the concrete (Gao et al., 2024).

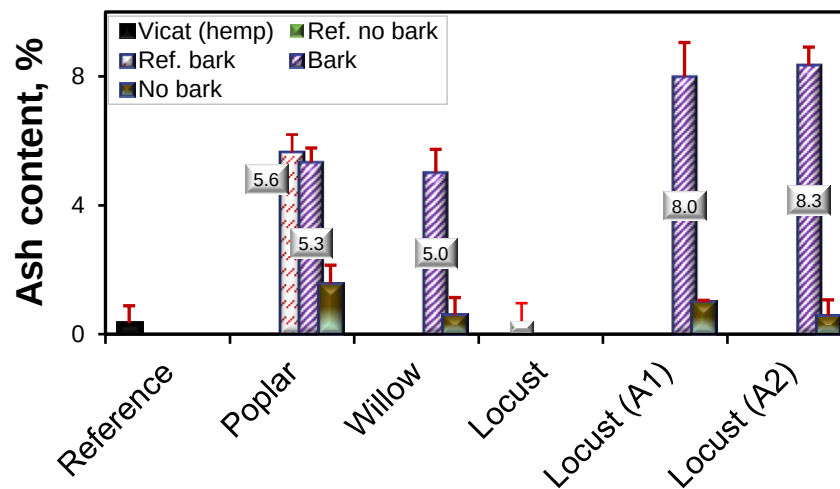


Fig 2.6. Ash content.

Inductively Coupled Plasma (ICP) analysis of elemental composition

The ICP analysis (Table 2.4) was used to determine the presence of heavy metals in the wood samples. For ICP, the reference samples are strictly compared based on whether they have bark or not, as opposed to samples from contaminated sites. The soil composition in Table 2.1 already proved evidence of the presence of such metals like Ni, Cu, Cd, Pb and Ar, which are also captured in the ICP analysis of the wood and in most cases, higher in the samples collected from contaminated soil.

The analysis clearly shows differences in elemental concentrations of the wood biomass and suggests some contamination in samples obtained from polluted soils with higher concentrations of heavy metals in most cases compared to the reference wood.

While soil analysis shows relatively high concentrations of chromium and lead for poplar and locust samples, we note that chromium content in wood poplar from contaminated soil (S4) is higher than reference poplar (S12) but lower in bark (S5) and (S6). Chromium content is also higher in black locust wood and bark (S8 to S9) compared to black locust reference wood (S14). Notably, locust wood is accumulating more levels of chromium than locust bark.

Lead contents are quite comparable between wood and bark poplar (S4 & S12; S5 & S13). For black locust, we can find a lower lead composition in reference samples (S14) than S8 and S10. S8 and S10 also accumulated more Pb than relative bark, but the variation in accumulation is rather high for locust wood.

Table 2.4. ICP analysis.

Wood	Sam.	Estimated concentration of metals in micrograms of the metal per gram of total solid, µg/g TS										TS, %
		Vanadium (V)	Chromium (Cr)	Manganese (Mn)	Cobalt (Co)	Nickel (Ni)	Copper (Cu)	Zinc (Zn)	Arsenic (As)	Cadmium (Cd)	Lead (Pb)	
Poplar	S12	<0.0056	0.07	12 ^(0.7)	0.036	<0.5	1.5 ^(0.1)	12 ^(0.6)	0.02 ⁽⁻⁾	0.43 ^(0.01)	17 ^(0.6)	96
	S13 (bark)	0.25	0.31	14 ^(0.9)	0.340	<0.5	4.2 ^(0.2)	89 ^(4.4)	0.14 ⁽⁻⁾	0.44 ^(0.02)	37 ⁽¹⁰⁾	96
	S4	<0.0053	0.26	6 ^(0.4)	0.029	<0.5	3.1 ^(0.8)	21 ^(2.9)	0.06 ⁽⁻⁾	0.31 ^(0.05)	8 ^(7.7)	96
	S5 (bark)	0.042	0.15	32 ^(2.7)	0.150	1.05 ^(0.1)	5.6 ^(0.2)	158 ^(6.9)	0.07 ⁽⁻⁾	0.33 ⁽⁰⁾	51 ⁽⁶⁾	96
Locust	S14	0.012	0.09	9 ^(1.2)	0.063	<0.5	1.9 ^(0.1)	4.3 ^(0.3)	0.04 ⁽⁻⁾	0.14 ^(0.1)	10 ⁽¹⁴⁾	97
	S8 (A1)	0.150	0.38	0.9 ⁽⁻⁾	0.010	<0.3	3.1 ^(1.1)	5.1 ^(1.3)	0.03 ⁽⁻⁾	0.17 ^(0.1)	34 ^(1.3)	97
	S9 (bark)	0.038	0.16	37 ^(2.5)	0.150	<0.3	4.5 ^(0.4)	184 ^(3.2)	0.05 ⁽⁻⁾	0.74 ^(0.01)	22 ^(2.5)	95
	S10 (A2)	0.008	0.71	2 ^(0.4)	0.007	<0.5	3.5 ^(0.8)	4.3 ^(0.7)	0.04 ⁽⁻⁾	0.15 ^(0.1)	45 ⁽²²⁾	95
	S11 (bark)	0.077	0.17	12 ^(0.3)	0.052	1.1 ^(0.2)	6.1 ^(0.4)	12 ^(0.4)	0.04 ⁽⁻⁾	0.14 ^(0.1)	21 ^(2.7)	96
Willow	S6	<0.0054	0.01	5 ^(0.5)	0.03	<0.3	3.0 ^(0.4)	27 ^(0.6)	0.03 ⁽⁻⁾	0.31 ^(0.01)	39 ^(13.5)	97
	S7 (bark)	0.027	0.08	17 ^(0.6)	0.21	0.55	4.7 ^(0.04)	156 ^(4.9)	0.04 ⁽⁻⁾	0.72 ^(0.04)	53 ^(8.2)	98

Dynamic vapour sorption (DVS)

The sorption behavior of the wood biomass appears to correlate with the trend in the ash content. Table 2.5 shows that reference Poplar (S12) presents the lowest moisture adsorption at 95% RH, followed by contaminated-soil samples of Locust-A2 and Willow. Compared to VH, the results in Table 2.5, including the sorption curves (Fig. 2.6), show that the wood samples without bark present lower hygroscopicity that is due to the generally higher degree of crystallinity in wood cellulose than that of hemp. The most substantial moisture adsorption can be traced to S9 (Locust A1 bark), which also correlates to high cumulatively (77%) amorphous content and high ash and extractive contents. The sorption hysteresis curves (Fig. 2.6) show that willow wood and relative bark are noticeably similar in moisture sorption behavior, despite the substantial amounts of inorganic content in willow bark. However, hysteresis shows a typical bark influence, acting as a barrier to moisture diffusion for the Locust samples. In the case of poplar, the results are similar to Willow, with no strong influence of bark or inorganic content. Although the trend suggests that wood from contaminated soil appears to adsorb more moisture, this is only apparent when compared to the samples without bark from uncontaminated soil. The most notable variation in hygroscopic properties is locust bark (S9) with the highest affinity for moisture.

Table 2.5. Equilibrium moisture content (EMC) of wood biomass is at 95% relative humidity.

	Vicat hemp	Poplar				Willow		Locust				
Code	VH	S1	S1	S4	S5	S6	S7	S1	S8	S9	S1	S11
EMC, %	22.1	18.7	21.1	21.6	21.9	19.7	19.6	21.3	20.1	26.2	19.4	22.4

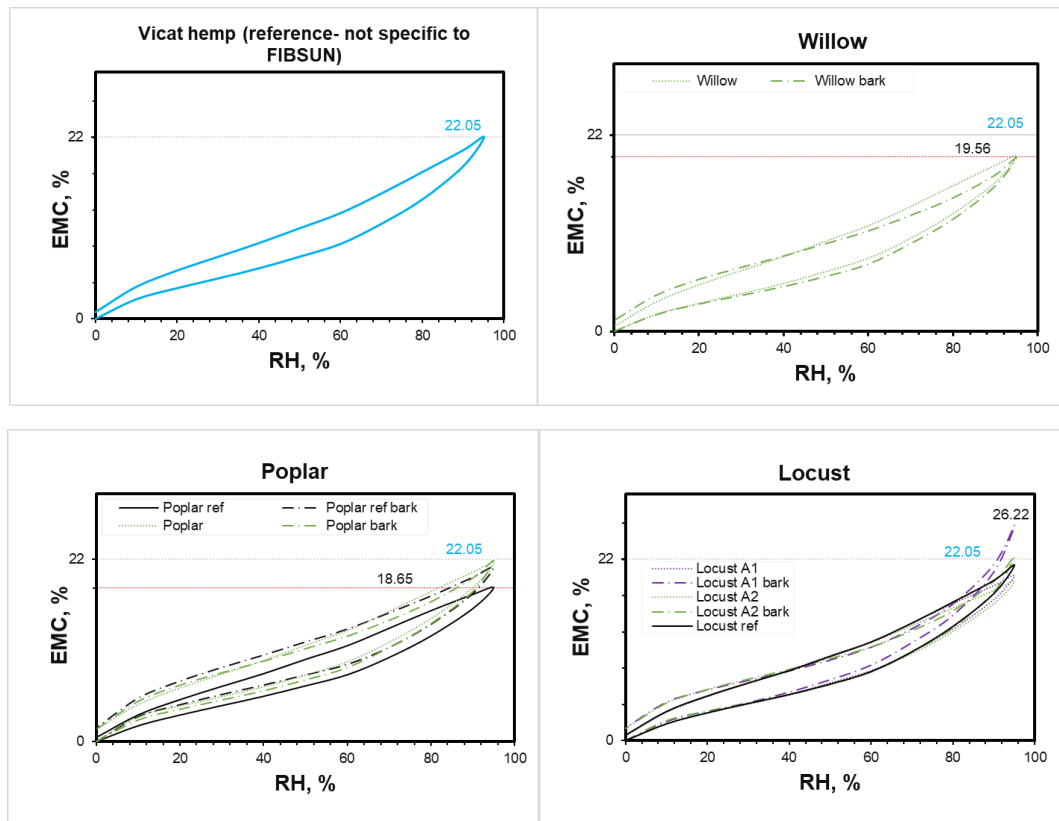


Fig 2.7. Moisture sorption hysteresis from DVS with a constant temperature of 25.4 °C and relative humidity (RH) 10 - 95%. Vicat hemp shive is also presented for comparison.

The pH was examined with 1 g (oven-dry basis) of each wood type mixed in 20 ml of distilled water (pH 6 ± 0.03). The samples were initially stored at room temperature, for at least 24 h before pH was recorded. Four (4) replicates were examined. Fig. 2.8 shows that the wood samples present a substantially more acidic pH than the reference Vicat (hemp) sample. When comparing the wood batches, the pH values were not so distinct. ANOVA Single Factor analysis with a confidence interval of 0.05 returned a p-value (p) of 0.05, which indicates that the results are exactly at the threshold of statistical significance. Initial pH measurements indicated no significant impact of contamination on poplar samples ($p > 0.05$). In contrast, the presence of bark significantly increased the pH of reference poplar samples ($p < 0.05$). For locusts, samples without bark from contaminated sites displayed significantly lower pH compared to reference barkless samples ($p < 0.01$), suggesting elements potential acidification due to heavy metals or organic contaminants. These correspond to the inorganic (ash content) measured for the samples. However, both reference barkless locusts and bark-containing locust samples from contaminated sites showed similar pH values ($p > 0.05$). Therefore, while the pH variation is suggestive, it is not definitive proof of influence of contamination, as factors such as microbial activity, inherent wood variability, and soil conditions can influence pH. No significant difference ($p = 0.13$) in pH was observed between bark-containing and barkless willow samples.

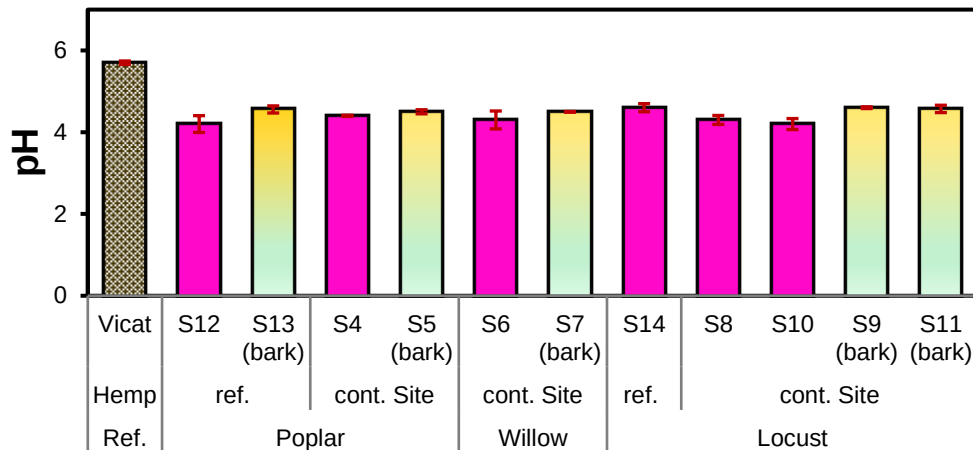


Fig. 2.8. Initial pH before buffering.

The pH buffering capacity of each wood type is examined and compared to the Vicat reference. The pH buffering capacity was measured by titration to a pH of 10 in an alkaline environment using 0.05 N sodium hydroxide (NaOH) solution. And to pH of 3 using 0.05 N hydrochloric (HCl) acid. Two (2) replicates were examined per buffering medium. Besides, after titration, samples from NaOH titration were left for over 72 h to evaluate the long-term release of reactive components in the wood substance. The pH was subsequently measured, and more base was added to reach the target pH. The pH buffering capacity is the number of moles of NaOH (or HCl) required to change the pH of the sample solution by 1 unit. It can also be obtained from the slope of the titration curve and mathematically by the weighted average buffering capacity in the mediums.

Fig. 2.8(a) presents the titration curves while Fig. 2.8(b) displays the average weighted total buffering capacity (buffering capacity in alkaline and acid environment). The titration curve for the Vicat reference (VH) is also presented. The results show that wood without bark contains lower average buffering capacity than hemp shives. This trend was synonymous with both alkaline and acid environments. This indicates that wood samples are more likely to resist neutralization by adding acid or base compared to the hemp reference. It can be observed that poplar from the contaminated site S4 and S5 have better buffering capacity than the sample from the sawmill. Bark samples show higher buffering capacity than wood and hemp shives. S5, S9 and S11 (bark from contaminated sites) exhibited the highest buffering capacity. The order of buffering capacity is shown below:

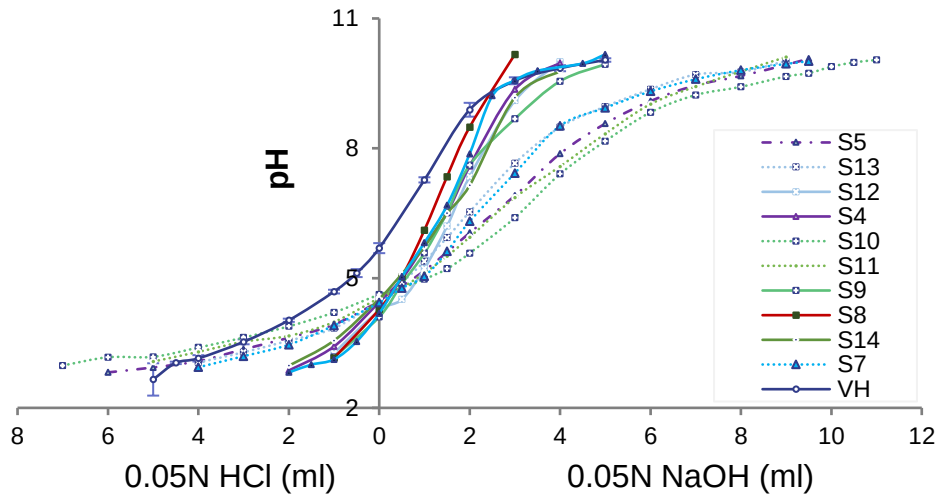
- **Poplar:** $S12 \leq S4 < \text{Vicat} < S13 < S5$.
- **Locust:** $S10 < S14 \leq S8 < \text{Vicat} < S9 = S11$.
- **Willow:** $S6 < \text{Vicat} < S7$.

The observed overall order of buffering capacity based on this result is such that:

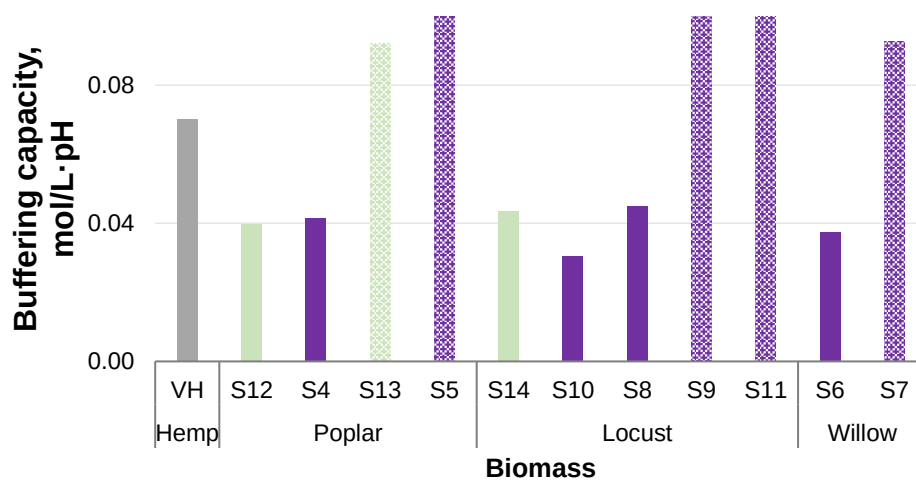
- $S7$ (Willow) < $S5$ (Poplar) $\leq$ $S9/S11$ (Locust).

This outcome indicates that bark has the highest buffering capacity, which aligns with the high lignin (see Tables 2.3), ash content Fig. 2.6 as well as the higher concentration

of heavy metals and minerals (Table 2.4), which can form complexes with organic compounds in bark, influencing pH buffering capacity. While there were slight differences between samples from contaminated and reference sites, the buffering capacity was generally similar, with a slight tendency for samples from contaminated sites to exhibit higher buffering.



(a)



(b)

Fig. 2.9. (a) Titration curves; (b) pH buffering capacity of all samples.

Fig. 2.10 shows the pH stabilization of the samples after 72 hours of storage. The final quantity of NaOH administered to re-titrate the samples to the target pH of 10 is also presented. Notably, there is a significant difference in the level of pH stabilization between the wood samples and the reference. The hemp reference experienced a substantial pH decrease to 5.8 ± 0.0 after 72 hours of initial titration to pH 10.05. Subsequently, cumulative titration to pH 10 was with 7 ml of NaOH. In contrast, most of the wood samples exhibited a pH drop of about 1.7 after 72 h, except for S7, which experienced the most significant pH decrease, dropping to 7.7, while S4 had the least

change, stabilizing at 8.6. So far, S9 shows the highest cumulative amount of NaOH titrant (14.5 ± 0.0 ml) to achieve a pH of 10 while S8 shows the lowest 4.0 ± 0.0 ml). The outcome somewhat follows the trend observed after the first titration, indicating substantial influence of bark on pH buffering capacity with S9 exhibiting the highest buffering capacity.

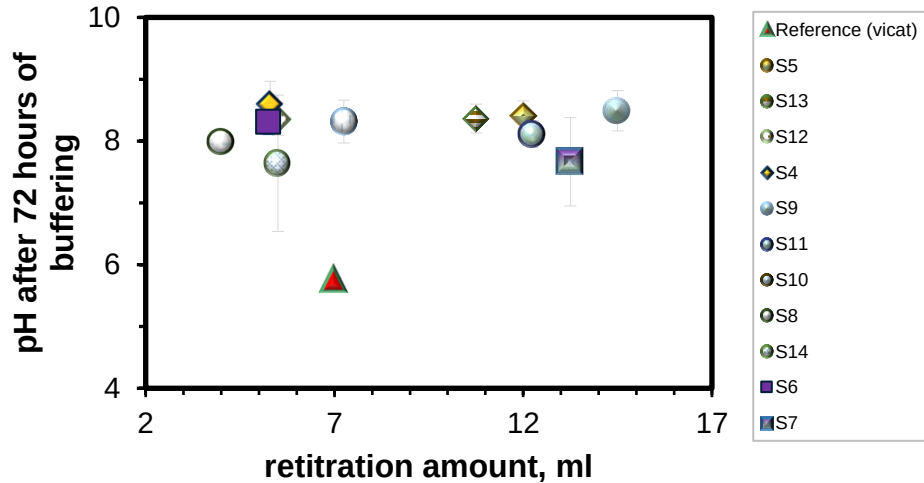


Fig. 2.10. pH of samples after 72 hours of buffering and relative amount of 0.05 N NaOH to re-titrate to pH 10.

Conclusions:

- Three wood species (Poplar, Locust and Willow) were harvested from a site (Grenoble, France) contaminated due to industrial activities from paints and varnish production.
- Poplar wood from the sawmill (S12), including its bark (S13), and locust wood (S14) were also examined as reference wood to ascertain the level of contamination and its impact on the composition and properties relative to the contaminated sample. Reference hemp shive (VH) was also characterized.
- ICP analysis revealed and confirmed absorption of heavy metals in the wood samples collected from the contaminated site. Most notably, Pb and Cd levels were rather high for these samples, particularly in the locust wood.
- Regarding pH, pH buffering capacity, DVS, and chemical composition, some variations were observed in the contaminated samples. However, these differences were rather intra-related (comparing wood with bark within a species) than when the species are compared based on contamination. For instance, pH and buffering capacity were more influenced by bark, due to the high mineral and extractive content. The implications of these variations were most significant when compared to the reference concrete material (hemp shive).
- Preliminary analysis indicates that the sorption behavior of the biomass harvested from the contaminated site aligns with that of the reference hemp shive. Though the locust bark S11 does show a notably high rate of water adsorption. Besides S9, the sorption characteristics of the wood biomass suggest their suitability in concrete, but the interplay between all other measured factors such as the level of metal contaminant and ash makes it crucial to determine the long-term effects on concrete performance and to confirm that the specific contaminants from the soil do not negatively influence concrete.

Recommendations for trials

The comparable sorption characteristics between the reference hemp shives and the wood samples from the contaminated site proposition their potential for use, even with moisture impact. The higher pH buffering capacity of bark also highlights potential benefits for compatibility with the Prompt Natural Cement (PNC) considered for trial validation in concrete applications (to be discussed in D2.3). However, it is pertinent to carefully select, thoroughly characterize, and consider other properties of wood fillers for the reliable production of the bioconcretes, especially considering the interplay between all factors. For instance, despite the high buffering capacity of bark, its high ash content implies a possible reduction in concrete compressive and flexural strength, while fire resistance might be improved. Moreover, the DVS analysis and levels of extractives in S9 do make it unsuitable for the desired application. The high levels of Pb and cadmium in locust wood is also a source for concern. Therefore, trials will focus on optimizing the wood additive formulation in relation to the measured properties. The results suggest utilizing poplar samples to achieve these, while comparative analysis can be prototyped with locust wood.



Cardoon (*Cynara cardunculus*)

Crop site and harvest description

The cardoon site is located in Ourique the hinterland of the Baixo Alentejo (South Alentejo) region, the long and extensive plains of Beja's district. The "Baixo Alentejo" region is characterized by vast expanse of low-relief, almost treeless, plains. Having high degrees of similarity among its profiles of soil occupation, and the physical characteristics of its territories leads it to be considered a homogeneous region.

Given the topography, soil characteristics (predominantly schistose and thin) and climate profile (typically Mediterranean), the land use in Ourique (Beja district) has always been associated with livestock and later to rainfed cereal crops (i.e. without recourse to irrigation). To take the most advantage of the soil, this farming practice evolved into a traditional culture rotation scheme, where after two years of cultivation of cereal lands are left in fallow (i.e. uncultivated) for three or more years, to recover fertility and serve as grazing for livestock (mainly sheep and cows).

Today, the extensive and plain surface of Baixo Alentejo is dominated by open and continuous fields where cereal fields, fallows, natural grasslands (meadows), and fields with leguminous plants prevail. With a little sloping relief, which is around 200 meters above sea level, this agricultural matrix is only interrupted occasionally by old olive groves and cork oaks, sporadic trees (alone or in small groups), known as Montado Landscape, patches of shrubby and/or arborous natural vegetation like cardoon.

The climate changes and edaphoclimatic conditions led to a scarce water level in this region, compromising agriculture practices and the needs of natural vegetation. The current water levels are around 180-190m below ground.

The cardoons produced are used for different purposes through a cascade valorization of the plant, including the flowers and seeds for food industry (common use) and stalk and leaves as source of fiber to produce a textile yarn. Two different harvests are needed as biomass harvesting is only allowed by producers after all the flowers and seeds have been collected. Such timelines are of great relevance, as the biomass valorization intends to add value to current productions, being that flowers and seeds are, as of today, the main economic output for cardoon producers, and the collection of biomass should not interfere with the main cardoon value chain. With that taken into consideration, the harvest of larger quantities of biomass for fiber extraction can only start in the beginning of June, which brings some challenges into consideration, such as the swift drop of moisture in the plants, and its consequent loss of fiber extraction viability, and the accurate harvest planning on such a thin timeframe. Since the site for pilot validation trials was not identified at the start of the project, the F4S partner had to identify and select a cardoon producer located on poor agricultural land and willing to participate in the project, with a commitment to provide stalk and leaf biomass harvested at an appropriate time. Moreover, the year 2024 had exceptionally hot and dry weather conditions, particularly before and during the harvest season of biomass, which led to an abrupt loss of quality and availability of biomass.

Two cardoon biomass harvests were made, firstly on the 17th of May 2024 where test samples were collected for initial plant and fiber characterization analysis (D2.2), and then on the 17th of July, a larger quantity for industrial testing of fiber extraction and yarn production. These constraints in biomass acquisition prevented the cardoon biomass



from being fully characterized in terms of its physicochemical properties. The following description of the plant is based on information documented in the literature.

Plant identification and description

Cardoon (*Cynara cardunculus* L.), also called Cynara or Castile thistle, is a plant of the Compositae (=Asteraceae) family, closely related to globe artichoke (*C. cardunculus* var. *scolymus* (L.) Fiori). Both varieties are included in the same botanical species (*Cynara cardunculus* L.), along with various wild types, and both are native to the Mediterranean basin. Both have been traditionally cultivated as vegetables in South Europe for centuries although for different crop products. Globe artichoke is grown for edible phyllary bases and receptacles of its floral heads, whereas the cardoon (*C. cardunculus* var. *altilis* (L.) DC) stalks are eaten, as these are the artificially bleached leaf rachises following high-input management techniques. Without such management, cardoon behaves as a hardy crop, featuring characteristics such as perennial life form, annual growing cycle, vigorous regrowth, bee-attractive flowers, and adaptation to drought stress, that make this plant a good candidate to be grown in the dry lands of the Mediterranean region as a perennial field crop for multi-purposes and non-conventional uses.

Cardoon can be described as a stout plant that can reach over 2m of height and often shows richly branched stems at maturity. Stems can be densely tomentose to subglabrous, with 14–44 ribs. Leaves are basally rosulate (basal leaves in a rosette) and densely set along the stem (cauline leaves), they are lanceolate in outline and variously pinnatifid, approximately 21–88 cm x 10–33 cm in size; the leaf base is wide (app. 30 mm), with marginal spines. Leaves exhibit from 11 to 25 segments narrowly ovate to ovate in outline, 29–170mm x 7–78 mm, narrowly triangular-lobed to serrate, apically caudate, with a yellowish spine. Leaf abaxial surface color ranges from pale greyish to whitish green depending on leaf indument (moderately to densely woolly); the adaxial surface is green, moderately pilose to glabrescent. There is a deep and fleshy taproot. Commercial varieties or cultivars of white cardoon usually have short - or do not have - leaf spines and leaf segments are often broader. Cynara is a perennial herb with an annual development cycle conducive to summer dormancy, a strategy of adaptation to adverse climatic conditions of that season in Mediterranean climates; thus, the above-ground plant parts die back in summertime, a period of severe drought, but the plant regrows in early autumn, when conditions are more favorable for plant growth. In botanical terms, plants with such a life pattern are called hemicryptophytes. Lifeform, development cycle and adaptation to Mediterranean climates are key criteria for the agronomy of this species as a field crop in drylands as well as for the feasibility of its applications.

At the end of the growth cycle, the aboveground biomass of Cynara is made up of basal leaves (rosette leaves), cauline leaves, stems and heads that bear oil seeds. The proportion of these plant fractions in the whole biomass or, in short, the biomass partitioning is important to the applications of this crop because a plant fraction can be more valuable than another for specific use e.g. seeds for oil or biodiesel while other plant fractions for thermal applications. Furthermore, biomass partitioning has a major effect on fuel properties due to differences in mineral composition of plant fractions.

Plant composition and properties



**Circular
Bio-based
Europe**
Joint Undertaking



Co-funded by
the European Union

This project has received funding from the European Union's Horizon Europe programme under grant agreement N° 101112318.

The biochemical composition of *Cynara cardunculus* is diverse and holds significant interest for various applications. Here, we present a detailed account based on available literature and scientific findings:

1. **Proteins and enzymes:** Cardoon flowers contain proteolytic enzymes such as *cardosins*, primarily cardosin A and cardosin B. These enzymes, extracted from the pistils of the plant, are glycoproteins with properties suitable for milk coagulation. Studies such as those by Veríssimo et al. (1996) have shown that cardosins possess high specific activity, with cardosin A playing a crucial role in cleaving κ -casein to initiate curd formation.
2. **Phenolic compounds:** Cardoon is rich in phenolic acids, including caffeic acid, chlorogenic acid, and cynarin. These compounds are known for their antioxidant activity, contributing to the plant's protective functions against oxidative stress. The presence of these phenolics is significant, with studies (e.g., Fratianni et al., 2007) demonstrating their ability to scavenge free radicals and potentially reduce risks associated with oxidative damage in biological systems.
3. **Fibers:** The lignocellulosic nature of cardoon makes it suitable for bioenergy production. The stems and leaves are composed of cellulose, hemicellulose, and lignin in varying proportions (approximately 30–35% cellulose, 20–25% hemicellulose, and 15–20% lignin). This composition renders it viable as a feedstock for second-generation biofuels, as highlighted by studies such as those by Ramos et al. (2013), which indicate its promising role in sustainable biomass production.
4. **Lipids:** Cardoon seeds contain oil with a high unsaturated fatty acid profile, including linoleic and oleic acids. This lipid profile has potential applications in both the nutritional and industrial sectors. Research has indicated that these fatty acids contribute to cardiovascular health when incorporated into human diets.
5. **Secondary metabolites:** Beyond phenolics, cardoon synthesizes other bioactive compounds such as sesquiterpene lactones, including cynaropicrin. Cynaropicrin has been extensively studied for its anti-inflammatory and hepatoprotective properties (e.g., Akashi et al., 2017), showing promise in pharmacological applications.
6. **Mineral content:** Cardoon contains significant amounts of essential minerals, including potassium, magnesium, and calcium. These minerals play vital roles in both plant growth and human nutrition, enhancing the crop's value for functional food applications.



Figure 3.1. Cardoon plantations

When the *Cynara* crop is intended for biomass or seed production, the best time of harvesting is summer, when the annual development cycle ends, the aboveground biomass has low water content, and seeds are ripe, but before seed dispersal and plant re-growth. In the Mediterranean region and depending on the crop location, the harvest time can be July, August, or September. Crop applications targeted for green biomass (to be used for biogas or forage) have been seldom reported, but it is worth noting that it will require different harvest timing. The choice of harvesting machinery depends on many factors, including the product to be harvested (e.g. dry biomass, seeds, lignocellulosic biomass or fresh biomass) and the availability of the machinery. *Cynara* whole biomass can be harvested by using a forage harvester mounted with a robust header (e.g. discs rotating counter directionally to each other); other options can be a drum mower followed by the baler, or a biomass harvester-baler like the Biobaler™. However, in order to optimize the exploitation of this crop, a separate harvest of the oil seeds and the lignocellulosic biomass (stalks, leaves, bracts and other heads parts) is advisable. The separated harvest can be achieved with conventional machinery in two steps by using a combination of harvester (to harvest the oilseeds) followed by a baler (to collect the lignocellulosic biomass). An alternative strategy is to harvest the whole biomass, take it to an ad hoc biomass facility and separate mechanically the different biomass fractions at the facility; in this way, each biomass fraction could be directed to different applications, following the biorefinery concept.



Figure 3.2. Cardoon plant and leaves

Scanning electron microscopy (SEM)

Morphological analysis of fibers is possible with SEM. This type of equipment is characterized by allowing the analysis of the diameter, uniformity, and roughness to the surface of the sample through its high resolution. The equipment used in the present work was the FlexSEM1000 II of Hitachi.

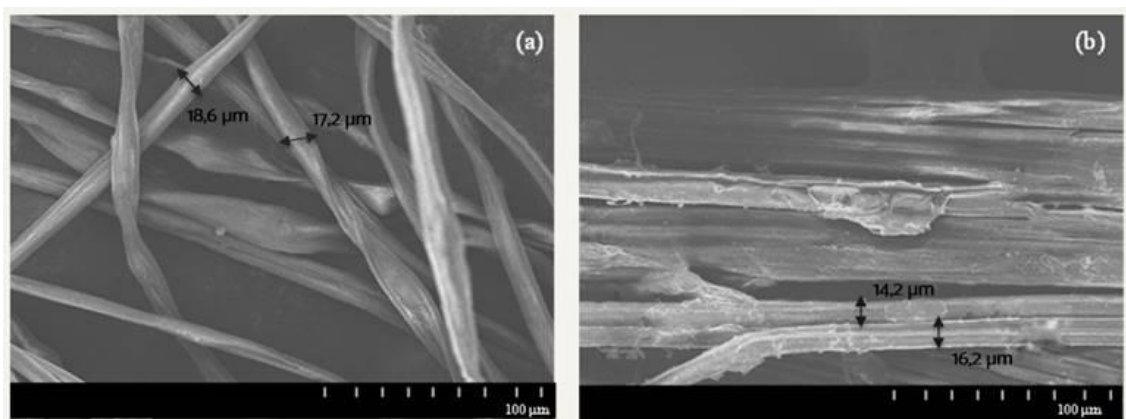


Figure 3.4 - SEM Images of the different fibers (a) cotton, (b) cardoon

SEM images provide excellent detail on the surface of the fibers. In the images represented in Figure 2.12 (a) and (b), we were able to observe the two fibers under study. In the case of Figure 2.12 (a), referring to cotton fiber, the presence of convolutions and inversion marks of direction of the spiral fibrillar structure along the length of the fiber can be observed. These characteristics have been demonstrated in the literature as unique characteristics of cotton and allow their easy distinction from other fibers. Regarding convolutions, they are twists that are often reversed along the length of the fiber, occur depending on the variety of cotton and are developed when the lumen fluid is lost, more appropriately in the drying stage. Marks of the reversal of direction of the spiral fibrillar structure appear during the fiber growth process in which the cellulose changes orientation in response to internal stresses or changes in plant metabolism.

In relation to Figure 3 (b), referring to the cardoon fiber, a longitudinal section of the fiber is observed in which it is possible to see some irregularities and roughness in the texture of the fiber surface, which may be related to the presence of impurities on the surface, not totally removed in the extraction process, which may also justify the irregularity of the fiber diameters along its length, which comprise values between 14.2 and 16.2 μm . Comparing the two fibers under study, they present some differences, the most notable being the lack of convolutions in the cardoon fibre, compared to those observed in cotton. Regarding the fibrils that are observed in the cardoon fibre, cotton can be linked to the growth and twisting of cellulose. Regarding the diameter, the cardoon fibre has a smaller diameter than cotton (17.2 and 18.6 μm) however the difference is not relevant

Fourier Transform Infrared Spectroscopy (FTIR)

To verify the similarities in the chemical composition of the two fibers, the FTIR method was used for each of the samples. The equipment used in this study was Bruker's Alpha II Platinum – ATR/Transmission at a wave number between 500 and 4000 cm^{-1} and with a resolution of 4 cm^{-1} . This equipment provides information on the bonds and chemical composition of the fibers, without prior sample preparation. The fibers only have to be inserted individually into the equipment ensuring that the entire crystal rests on the sample, in the end, the results are obtained from a transmission graph (%) as a function of the wavenumber (cm^{-1}).

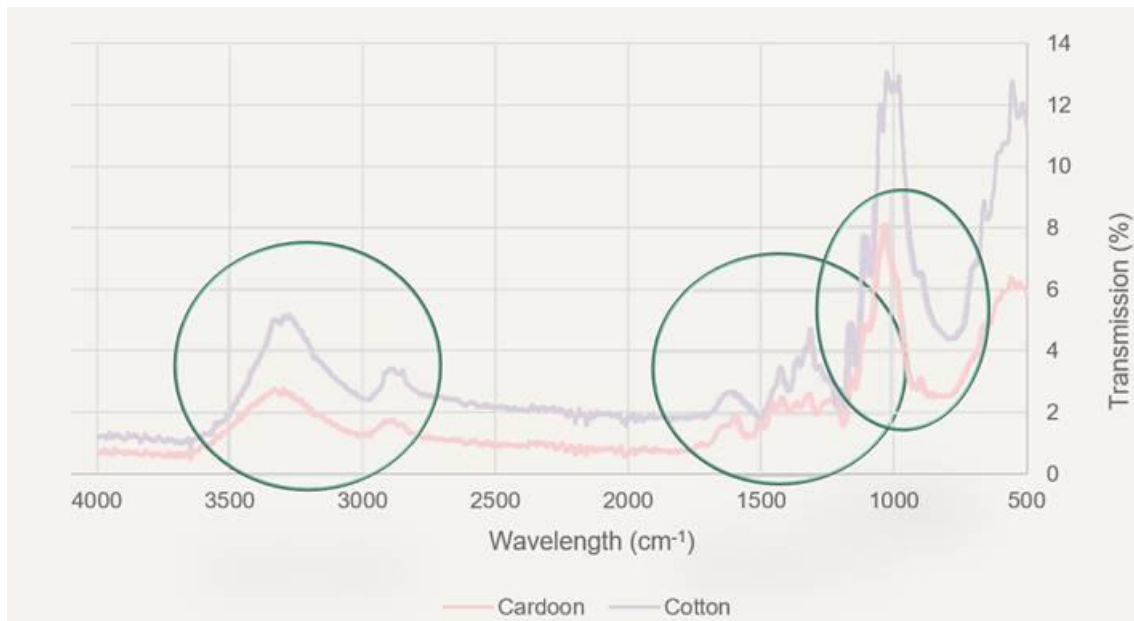


Figure 3.5 - FTIR spectrum

The FTIR spectrum was analyzed in the 4000-500 cm^{-1} region, covering the characteristic bands of the main constituents of plant fibers: cellulose, hemicellulose and lignin. These components are mostly made up of alkanes, aromatic compounds, alcohols, esters, and ketones with functional groups that contain oxygen. By analyzing the curves, it is possible to identify several absorption peaks characteristic of natural fibers, highlighting the first two bands in the intervals of 3400-3250 cm^{-1} and 2900-2800 cm^{-1} . The FTIR spectrum obtained shows striking similarities with the spectrum of cotton fibers described in the literature, evidenced by absorption bands in the intervals 3400-3250 cm^{-1} , 2900-2800 cm^{-1} , 1650-1550 cm^{-1} , 1500-1250 cm^{-1} and 1100-970 cm^{-1} . It is found that cardoon fiber also has these absorption bands.

These bands are attributed to different vibrations of bonds between atoms in the molecules. The absorption band between 3400-3250 cm^{-1} corresponds to the elongation vibration of the 12 group OH-, indicating inter- and intramolecular hydrogen bonds. The band between 2900-2800 cm^{-1} is associated with CH group vibrations, due to the presence of the CH₂OH group in cellulose. The absorption band between 1650-1550 cm^{-1} refers to the elongation vibration of the unconjugated C=O groups of hemicellulose.

In the case of the cardoon curve, two peaks are identified in this interval, one close to 1580 cm^{-1} , attributed to the elongation vibration of the unconjugated C=O groups of hemicellulose, and another close to 1620 cm^{-1} corresponding to the elongation vibration of CO conjugated groups present in lignin. In the range of 1500-1250 cm^{-1} , several bands are observed in both fibers. The band around 1500-1400 cm^{-1} corresponds to the vibration of the CH₂ group in cellulose, while the band between 1350-1250 cm^{-1} is attributed to the symmetrical and antisymmetric elongation of the CH groups in cellulose and hemicellulose, respectively.

Bands between 1100-970 cm^{-1} indicate the antisymmetric elongation vibration of the C-O-C bonds in cellulose and hemicellulose. Additionally, a peak close to 900 cm^{-1} is

observed, associated with the C-H functional group present in lignin. These peaks show the presence of functional groups in cardoon fibers, associated with the content of cellulose, hemicellulose and lignin, which are also the main components of lignocellulosic fibers. However, some degree of differentiation is observed between the fibers, and it is evident that cotton contains a higher amount of cellulose and hemicellulose compared to cardoon. On the other hand, cardoon has more impurities, translated by a greater number of peaks in regions where the main constituents are not present. This difference may be related to the process of extraction of cardoon fiber, since, as observed in the SEM analysis, the surface of the fiber presented impurities and irregularities.



**Circular
Bio-based
Europe**
Joint Undertaking



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(*Typha sp.*) and Reed (*Phragmites australis*)

Crop site and harvest description

Cattail was harvested from a rewetted peatland (3.2 ha) in Ankeveen the Netherlands, which was formerly used as a low production agricultural grassland (Fig. 4.1). The harvest was conducted using an electrical brush cutter (STIHL FSA 120). To observe seasonal differences in crop quality, cattail was harvested in four different times: November 2023 (autumn harvest), January 2024 (winter harvest), May 2024 (spring harvest) and August 2024 (summer harvest). A small amount of Cattail biomass was sampled also in April for protein analyses. Another test harvest was also done in August 2023 in Helmond, a different location in the Netherlands. Flowers were not separated from the rest of the plant.



Fig. 4.1. Area view of the *Typha latifolia* harvesting site in Ankeveen, NL. Photo: Cynthia Schild, Waternet

Common reed cultivation is studied at a 4-ha rewetted peatland site in Kauhajoki, Finland. However, this site is not productive enough for the needs of FIBSUN PVTs, and the material for the analyses and processing was bought from a commercial supplier that harvested it from a lake in southern Finland in March 2024.

Plant identification and description

Typha latifolia, commonly known as broad leaf cattail, is a wetland plant found in temperate and subtropical regions (Figure 3.2). It typically grows to an average height of 1.5 to 3 meters. The stems are cylindrical, with an average diameter of 1-2 cm. However, in nutrient-rich environments, the diameter can sometimes be slightly larger. Cattail takes about 2-3 years to reach full maturity, depending on the environmental conditions (light, moisture, nutrients). Once mature, it produces distinct brown, cigar-shaped flowers, which develop from the upper part of the plant. A single plant can live for several years, with new shoots emerging from the rhizome each growing season.



Fig. 4.2. *Typha latifolia*, broadleaf cattail (left) and *Phragmites australis*, common reed (right).

The plant has a high-water content (approx. 60%) and is relatively dense compared to many other wetland species. A full grown *Typha latifolia* plant can weigh anywhere from 0.5 to 1 kg when fresh, but its weight significantly decreases once it dries out, as it is highly moisture dependent.

Cattail is a resilient species and usually shows little physical damage unless affected by extreme environmental factors such as heavy storms, ice, or herbivore feeding. *Typha latifolia* is also very disease resistant but occasionally may be affected by fungal infections.

Common reed, *Phragmites australis*, is an aquatic plant in the grass family Poaceae (Fig. 3.2). It is a wetland grass that is typically 2-4 m tall and is distributed worldwide. It is common in both alkaline habitats and brackish water. *Phragmites australis* commonly forms extensive stands, reed beds, which may be as much as 1 km² or more in extent. Where conditions are suitable, it can also spread to 5 meters or more per year by horizontal runners. It can grow in damp ground, in standing water up to 1 m or so deep, or even as a floating mat. The leaves are 18–60 cm long and 1–6 cm broad. The flowers are produced in late summer in a dense, dark purple panicle, about 15–40 cm long. Later the flowers appear greyer due to the growth of long, silky hairs.

Methods

The cattail and reed were characterized based on their physical and ultrastructural properties, chemical composition analysis (lignin, cellulose, hemicellulose content) done using the Van Soest protocol, crystallinity assessment using X-ray diffraction (XRD) and thermal analysis via thermogravimetric analysis (TGA).

The nitrogen (N) content in cattail biomass, (whole plant and fiber) and in *Typha* juice (side product of the fiber extraction) were determined from 4-5 mg of each dried and milled biomass fraction with an elemental CNS analyser (Vario MICRO cube; Elementar Analysensysteme, Langenselbold, Germany). Protein content was calculated based on the N content using Kjeldahl method.

Composition and properties of the cattail and reed fibers

Morphological characteristics

Particle size analysis with QICPIC: The fiber size distribution and summary results (mean and median fiber size) of cattail fibers, as analyzed using QICPIC, are similarly presented in Fig 3.3 and Fig 3.4, respectively. This analysis was conducted to investigate the physical properties of the fibers following protein extraction (see D2.2). For reed, particle size analysis is detailed in **D2.2** report, as this was examined to optimize the blade gap opening for achieving the desired physical morphology similar to the conventional biomass used by SOPREMA in fabricating thermal insulation boards. While there were slight differences in the highlighted results, the differences in width and length were not notable (p -value = 0.141 and 0.132, respectively), regardless of harvest. The major change was found for winter harvest, where the fiber width after freeze-drying was found to be substantially lower (p -value = 0.006) than the unfrozen variant. Summer-harvested cattails are being analyzed, while spring cattails were found to be excessively long and incompatible with QICPIC analysis. A separate analysis tool, based on Fibershape/sieve analysis, is currently being developed to supplement the results.

The results in **Fig. 3.3** show the seasonal impact on cattail fiber length. Two batches of fibers were examined per season: non-frozen (NF) and frozen (F). It can be observed that spring and summer harvest fibers are longer than autumn and winter batches. Additionally, the autumn and winter fibers also present the highest amounts of fines (median (**Fig. 3.4**) = cumulative distribution % at 50%). Images provided in **Fig. 3.5** corroborate this finding, highlighting visible differences in fiber lengths, with spring and summer fibers appearing noticeably longer than fall (autumn) and winter fibers. The images also suggest a high number of fines in the latter, consistent with the fiber analysis.

In view of the impact of freezing, the winter harvest was more significantly affected, with a notable decrease in fiber length and a high number of fines in winter_F (**50% of fibers < 2381 μ m**). This represented approximately 17% more shorter fibers compared to the winter_NF batch. Freezing was found not to affect fall (autumn) fibers. The corresponding increase in fines for spring and summer harvests with the frozen batch was 10% and 11%, respectively.

The fiber lengths of the autumn cattails are 1.5 to 2 times shorter than the alternative wood fibers from SOPR (fiber length = 8000 μ m). These shorter fiber lengths may affect the ability to achieve the desired dimensional and compaction targets for the thermal insulation panels. While the slightly long Typha fibers (spring and summer) could be more expedient in this regard.

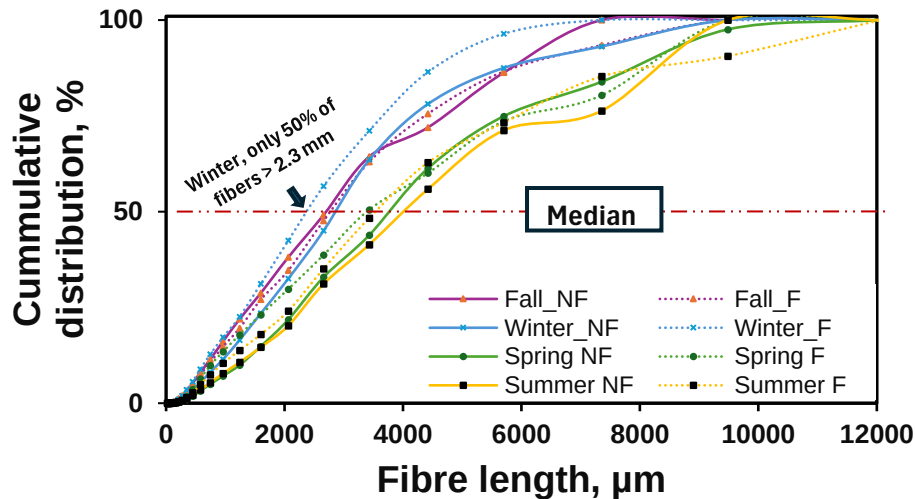


Fig 3.3. Particle size distribution of cattail after protein extraction (F (Frozen) and NF (Unfrozen)).

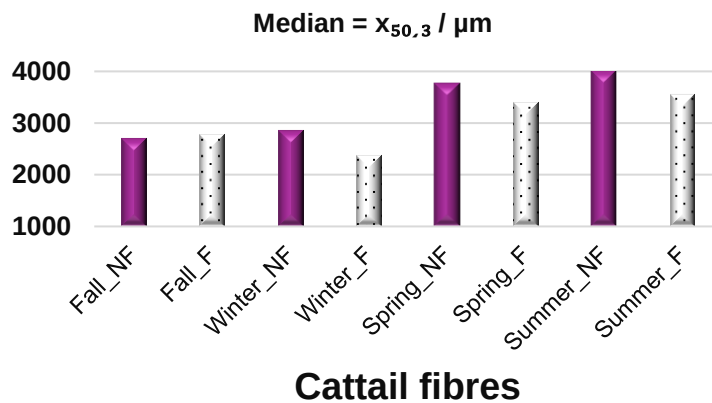


Fig 3.4. Median size.

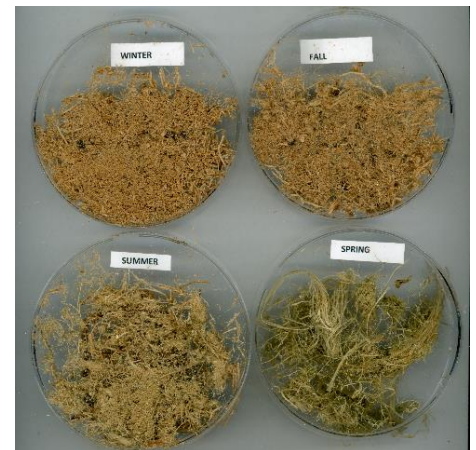


Fig 3.5. Cattail fibers from various seasons (spring and summer harvest appear longer with less fines).

Chemical Composition

Table 3.1 compares the **chemical composition** of cattail, reed, and SOPR reference wood. All cattail samples included flowers; however, in the case of the May harvest, the flowers were not yet fully developed. Frozen cattail was found to have slightly lower levels of soluble compounds and hemicellulose compared to unfrozen cattail, but it exhibited a higher residual ash content. This outcome is also supported by TGA analysis. Lignin, an important component affecting thermal insulation performance, is lower in cattail and reed than in wood. Overall, summer-harvested Typha shows the highest cellulosic content and the lowest hemicellulose and lignin content. The soluble content was found to be the lowest in winter and spring harvests. Freezing did not appear to impact the composition of the fibers.

Table 3.1. Compositional analysis of reed and cattail.

Biomass	Samples	Soluble content, %	Hemicelluloses, %	Celluloses, %	Lignin, %	Residual ash, %
Wood	SOPR reference	7.6 ± 1.8	14.7 ± 0.9	48.3 ± 3.1	25.8 ± 1.6	6.1 ± 1.6
Typha (Cattail)						
Autumn (fall) harvest (November)	NF	22.2	11.1	53.5	12.1	1
	F	22.2 ± 1.5	11.4 ± 9	62.2 ± 3.5	9.6 ± 3.6	0
Winter harvest (January)	NF	12.8 ± 0.8	23.8	59.2 ± 10.1	11.1 ± 1.6	0 – 2
	F	15.2	21.2	49.5	12.1	2
Spring harvest (May)	NF	17.1 ± 2.5	13.1 ± 0.5	47.5 ± 9.1	20.5 ± 8.2	0 - 5.1
	F	15.7 ± 3.3	20 ± 5.7	43.7 ± 9.4	13.8 ± 6.3	2.2 - 11.7
Summer harvest (August)	NF	22.3 ± 2.1	5.5 ± 0.6	64.2 ± 1.1	7.1 ± 1.2	0 - 2
	F	21.2 ± 3.8	3.8 ± 0.8	60.9 ± 1.5	8.7 ± 0	4.4 - 6.5
Reed	Without flower	4.6	14.0	56.8	14.8 – 28.6	2.8
	With flower	11.6	16.5	52.4	11.3	7.2

The **XRD** analysis is presented in Fig 3.6 and Table 3.2.

The XRD diffraction patterns show that reed has cellulose crystallographic planes (I_{002} , I_{101} , and I_{010}) like SOPR wood reference, which are different from cattail samples. The (002) plane is associated with cellulose chain stacking, typically appearing at around $2\theta = 22-23^\circ$. The (101) and (010) planes represent cellulose chain packing and lateral packing, respectively, and can vary depending on the cellulose structure. This indicates differences in the cellulose structure between the reference wood and cattail. However, the arrangements for the cattail variants are similar.

The crystallinity index (I_c) in Table 3.2 indicates the same trend, with the reference wood having a significantly higher I_c than any cattail sample. Among the cattail samples, the **summer harvest** presents the highest I_c . Although a variety of factors (including chemical composition, the density of the solid cell wall) play a role in the thermal conductivity of a lignocellulosic biomass, a higher I_c typically means increased thermal conductivity for a lignocellulosic material; therefore, the cattail samples appear to be better in this regard, with summer potentially being more thermally conductive. There was a minimal decrease in I_c with freezing for autumn and spring harvested Typha, but the I_c of winter and summer Typha increased notably after freezing. Of all cattail samples, autumn-harvested cattail (**November**) consistently showed the lowest I_c .

The I_c of reed, with and without flower, remained unchanged. Therefore, the use of reed with flower will not affect thermal conductivity in terms of I_c .

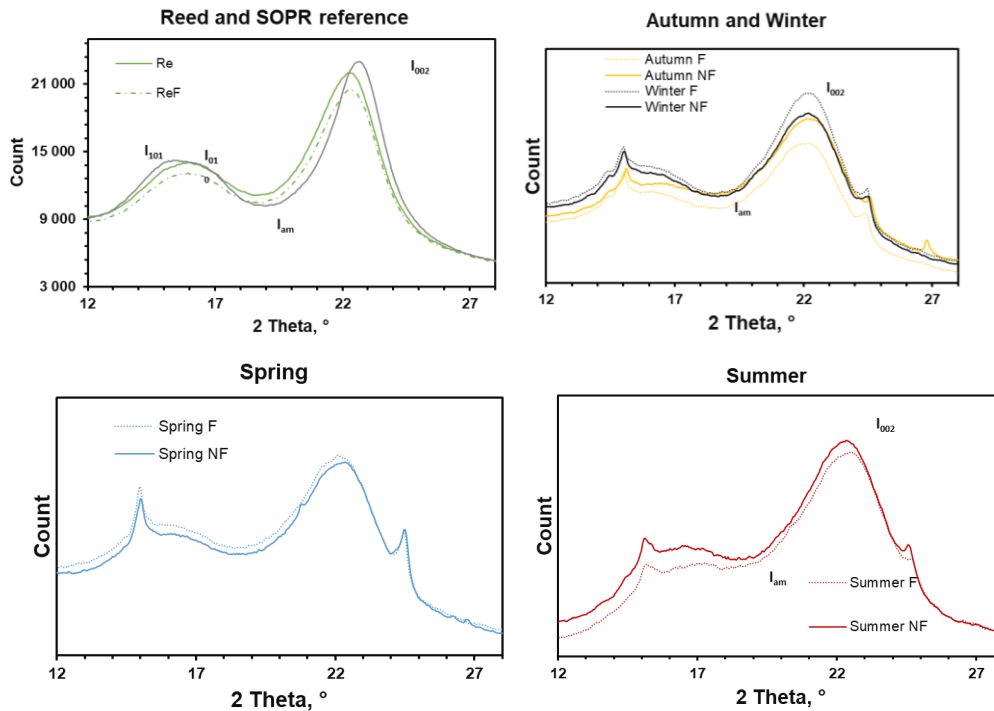


Fig 3.6. X-ray diffraction (XRD) pattern.

Table 3.2. Crystallinity Index (I_c).

Type	Material	Crystallinity index (I_c), %
Reference	Wood	55.50
Cattail - Non frozen	Autumn harvest (November)	34.82
	Winter harvest (January)	37.34
	Spring harvest (May)	35.03
	Summer (August)	40.82
Cattail - Frozen	Autumn harvest (November)	33.66
	Winter harvest (January)	41.34
	Spring harvest (May)	34.30
	Summer (August)	43.66
Reed	No flower (Re)	49.50
	with flower (ReF)	49.00

The TGA mass loss curves (Fig. 3.7) indicate that the thermal degradation behavior of cattail varies with the season. These differences could be attributed to seasonal changes in chemical composition, as already shown in the chemical composition and XRD results. This difference in cattail's thermal stability compared to the SOPR reference wood is summarized with the TGA data presented in Fig. 3.8.

The temperature at 5% mass loss indicates the initial thermal stability of the materials and is related to the decomposition of moisture and volatile compounds. The temperature at 10% mass loss indicates the decomposition of less stable components like hemicelluloses. While the overall thermal stability of the materials is highlighted by the temperature at 50% mass loss, this crucially reflects the degradation of major cellulose components. Higher temperatures for these points generally indicate greater thermal stability of the material. Therefore, the SOPR wood reference is notably the most thermally stable, with its 50% mass loss occurring around 360-370 °C (p-value < 0.05).

This range is typical for wood, due to a combination of high lignin content and more crystalline cellulose (already highlighted by XRD).

Although there was no substantial difference among the cattail fibers, **Typha Summer harvest** appears to have the highest thermal stability among the Typha samples, reaching 50% mass loss at a temperature comparable to or slightly higher than other Typha seasons (**around 328-340 °C**). This aligns with the previous insight that summer-harvested Typha has a higher I_c , with more cellulosic content and the lowest hemicellulose and lignin content, as cellulose typically degrades at higher temperatures than hemicellulose.

Autumn harvests show **notably (p-value < 0.05)** higher initial degradation temperatures at 10% mass loss, despite having a substantially lower (p-value = 0.004) initial decomposition temperature at 5% mass loss than spring and summer harvests. This behavior highlights the increased stability of primary components following the rapid loss of the very volatile fraction.

The Typha samples generally have higher residue/inorganics content (%) than the Wood reference, though this difference was not significant (p-value = 0.53). Ultimately, even though the summer harvest contained the highest amounts of inorganic residues (25-31%), the insignificant difference should not pose a concern with regards to achieving the absolute lowest thermal conductivity. On the positive side, these inorganics could potentially be beneficial for flame retardancy and dimensional stability at higher temperatures.

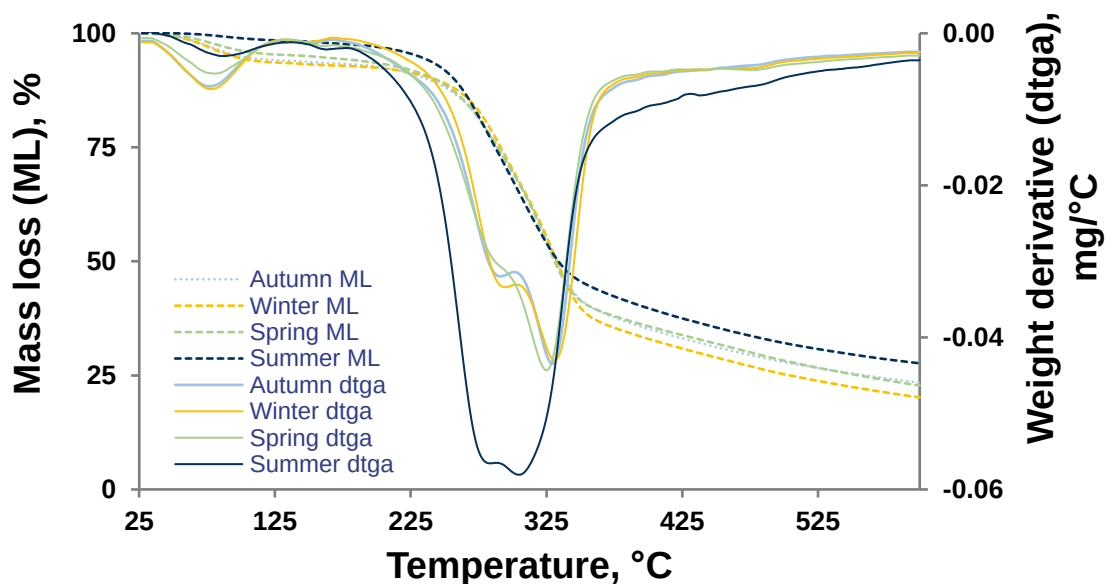


Fig 3.7. Thermogravimetric analysis of the Cattail (*Typha spp.*) samples.

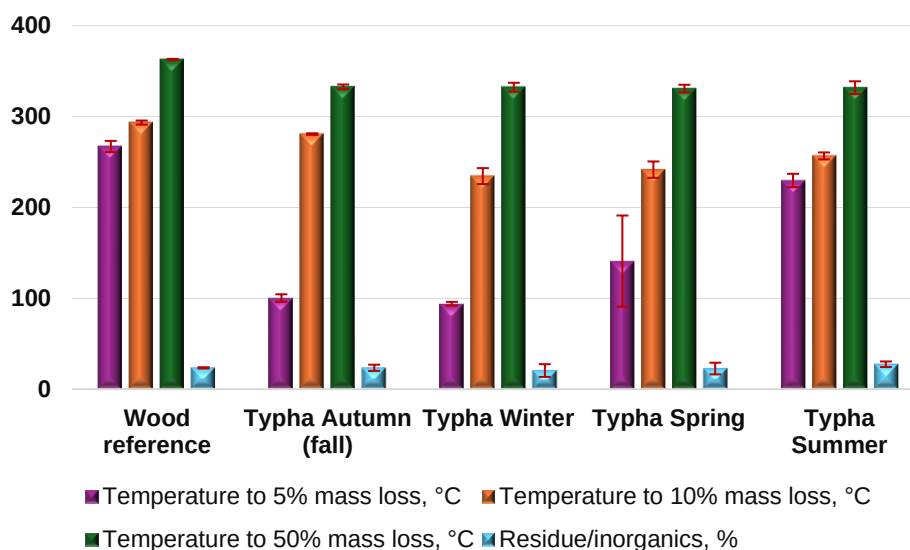


Table 3.4 compares the protein contents in Typha biomass (whole plant and fiber) and in Typha juice (side product of fiber extraction) between different harvest times. In August 2024, two kinds of biomass were collected: plants that were only cut once after growing the whole season (1st cut) and regrowth from May 2024 harvest (2nd cut).

Table 3.4. Nitrogen (N) and protein % in Typha latifolia biomass in 3 fractions: whole plant, fiber and Typha juice.

Harvest	Cut	Type	Season	Location	N %	Protein % DW	Protein g/L for juice
1-08-23	1 st	Typha juice	Summer	Helmond	3.13	36.1	13.9
	1 st	Fiber	Summer	Helmond	1.02	6.4	-
1-11-23	1 st	Whole plant	Autumn	Ankeveen	0.84	8.2	-
	1 st	Typha juice	Autumn	Ankeveen	1.48	28.1	3.1
	1 st	Fiber	Autumn	Ankeveen	0.66	4.1	-
15-01-24	1 st	Whole plant	Winter	Ankeveen	0.84	5.2	-
	1 st	Typha juice	Winter	Ankeveen	1.37	29.9	1.0
	1 st	Fiber	Winter	Ankeveen	0.55	3.4	-
26-4-24	1 st	Whole plant	Early spring	Ankeveen	2.32	14.5	-
14-05-24	1 st	Whole plant	Spring	Ankeveen	1.98	12.3	-
	1 st	Typha juice	Spring	Ankeveen	2.28	35.3	7.1
	1 st	Fiber	Spring	Ankeveen	1.68	10.5	-
1-08-24	1 st	Whole plant	Summer	Ankeveen	1.04	6.5	-
	1 st	Typha juice	Summer	Ankeveen	1.35	31.9	4.2
	1 st	Fiber	Summer	Ankeveen	1.13	7.1	-
	2 nd	Whole plant	Summer	Ankeveen	1.41	8.8	-
	2 nd	Fiber	Summer	Ankeveen	1.45	9.1	-

Based on the results, protein contents in the dry matter of whole plants are the highest in the early spring with 14.5% and lowest in the winter with 5.2%. Interestingly the *Typha*

juice from the harvest sampled in August 2023 in Helmond had the highest protein content, 13.9 g/L. In Ankeveen the highest protein content in *Typha* juice was recorded in May 2024 with 7.1 g/L and lowest in January 2024 with 1.0 g/L.

Conclusions:

- Cattail biomass, harvested in different seasons, and reed fibers including fibers and flower mix have been detailed.
- The extraction of protein from harvested cattail has been reported, with harvest seasons impacting the obtained protein content.
- The biomass remaining (fibers) from autumn, winter and spring harvest, after extraction of *Typha* juice have been studied and characterized through, particle size analysis (autumn and winter), composition analysis, thermal analysis and crystallinity studies, indicating some slight variations in properties with biomass and season of harvest.

Recommendations for trials

The characterization of reed biomass is indicative.

The analysis conducted on the cattail harvest and fibers from the different seasons shows some notable results. Based on the outcomes from fiber characterization, the long fiber length and thermal stability of the summer harvest as well as high protein content in spring harvest, show promise in achieving the desired final outcomes for the FIBSUN project. Regardless, the lab validation trial will first consider analyzing the fibers obtained from various harvests and correlate the performance of the prototypes to the fiber properties to achieve final product optimization for industrial viability.

Overall conclusions

Data on the quality of crop materials have been used for underdamping their potential for use in lab scale products (different PVTs), and in some cases for providing feedback in harvesting/ processing for optimum fiber generation technologies.

The work on crop characterizations will continue in the coming period in close exchange with each PVT as we will be moving from lab to pilot scale. Properties of crop materials play an essential role in product performance and therefore will always need to be monitored/ controlled and improved.

