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D3.1 REPORT ON FEEDSTOCK PRE-TREATMENT

pHYBi
Phytomanagement for a bio-based textile industry

PHYtomanagement as a sustainable feedstock source of lignocellulosic-based high-value BIo-based products for textile applications

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EXECUTIVE SUMMARY

The D3.1 Report on Feedstock Pre-Treatment describes the processes used to prepare poplar biomass as part of the pHYBi project, which aims to convert lignocellulosic biomass into high-value bio-based products, especially for textile applications.

This report is part of WP3 and summarises the activities carried out under Task 3.1 through a collaborative effort between PHY and CETIM. This document details how the poplar biomass underwent several pre-treatment stages. Debarking methods were evaluated in a literature review and the investment in mechanical debarking machinery was shown to be economical for processing upwards of 2 tonnes of biomass. For the small-scale samples generated in this task, the biomass was manually debarked and then air-dried, reducing its weight and preventing mould during transport. Following this, the material was dried in a controlled environment to remove moisture and avoid degradation. The biomass was then shredded in multiple stages to achieve a uniform particle size, which enhances its suitability for further processing. While a finer milling stage was tested, it was ultimately not used due to material loss concerns.

As a result of these processes, the biomass was standardized with consistent moisture levels and particle size, creating optimal conditions for subsequent steps like organosolv fractionation and valorisation. The established protocols ensure reproducibility and scalability for pilot-scale operations.

The pre-treated biomass will now be used in the next phase of the project, which includes organosolv fractionation and hemicellulose fermentation for producing pigments.

1. INTRODUCTION

1.1. DESCRIPTION OF THE DOCUMENT

The transition from raw agricultural feedstock to high-value industrial materials requires a precise bridge between field operations and laboratory refinement. This process is rooted in a circular economy approach that pairs soil remediation with biomass valorisation, turning crops grown on contaminated or saline lands into sustainable inputs for the textile industry. The primary objective is to demonstrate that industrial crops can serve the dual purpose of improving the soil while providing the raw building blocks, cellulose, lignin, and hemicellulose, which are needed for advanced bio-based products and textile coatings.

To achieve this, raw biomass must undergo a rigorous physical pre-treatment to eliminate natural variability and ensure the material is in an optimal state for chemical and biological processing. Standardizing the feedstock is critical; without uniform moisture levels and particle sizes, downstream extraction and fermentation processes cannot achieve the reproducibility or efficiency required for market-ready applications.

This report documents the methodologies and results associated with the initial preparation of poplar biomass. It details the transition of raw trunks and branches into a standardized, laboratory-ready feedstock, covering the mechanical debarking used to separate wood from bark and the thermal drying protocols established to stabilize the material against microbial degradation. Furthermore, the document describes the multi-stage shredding techniques employed to achieve a precise granulometry.

The primary output of this delivery is a validated pre-treatment protocol that ensures a consistent moisture content and a specific particle size of 0.5 mm. This particle size was determined to offer the best balance between the maximisation of surface area and the minimisation of loss from processing. These standardized characteristics are vital for subsequent stages, such as organosolv extraction and hemicellulose fermentation, as they maximize reagent accessibility and experimental accuracy. By establishing these foundational steps, the work confirms that the harvested biomass is conditioned to undergo efficient fractionation, ultimately supporting the transformation of remediation crops into sustainable innovations.

1.2. WPS AND TASKS RELATED WITH THE DELIVERABLE

This deliverable refers to Task 3.1 (Feedstock pretreatment) included in WP3: Feedstock valorisation: Extraction and characterization of products. The results of this deliverable are further used in T3.2 and T3.4 to optimise the extraction process and valorise the resulting hemicellulose fraction.

2. METHODOLOGY

Task 3.1 was initiated with a literature review from both PHY and CETIM on the economics and feasibility of different debarking methods and the influence of particle sizes and pre-treatments on the extraction efficiency.

The methodology implemented in Task 3.1 can be summarised in the following stages:

1. Harvesting and separation of biomass fractions (trunks and branches).
2. Transfer of material to PHY
3. Debarking of trunk biomass, evaluation of efficiency and cost-effectiveness of different methods.
4. Preliminary air drying to minimise microbial degradation during transport.
5. Transfer of material to CETIM
6. Controlled oven drying to constant weight.
7. Sequential particle size reduction and homogenisation.
8. Evaluation of final particle size suitability for downstream extraction and compositional analyses.

Regular coordination between PHY and CETIM ensured traceability of the biomass batches, harmonisation of processing conditions, and alignment between feedstock preparation and the requirements of subsequent extraction and fermentation activities developed in Tasks 3.2 and 3.4.

3. DEBARKING OF POPLAR TRUNKS

Literature review showed three feasible methods for debarking poplar trunks generated by UMLP: manual debarking of trunks using a peeling knife, mechanical debarking using a debarking machine and separation of bark from wood chips via flotation.

Flotation methods to separate bark from wood chips were investigated and two patents for machines were found (US3032188A and US4140281A), but no commercial supplier of a suitable machine could be located. In general, chipping the samples before debarking significantly reduces the purity of the obtained biomass and adds moisture, both of which may cause problems in the organosolv process, so this method was discarded and focus was shifted towards manual or mechanical debarking of trunks.

Mechanical debarking is typically performed on large de-branched trunks and can be a very quick and effective method for achieving a good wood quality for chipping. Manual debarking achieves the same results using a slower, more work-intensive process, but can be cost-effective for small scale samples, as it does not require a large upfront investment. At an estimated investment cost of 10.000€ for a debarking machine, the economical break-even point was estimated to be a weight of around 2 tonnes of tree trunks (Table 1). Since the samples sent by UMLP were small, the manual debarking method was applied. We estimate that for future samples below 200 kg, manual debarking will continue to be applied, whereas a debarking machine could be rented for sample sizes above this weight.

Table 1. Estimations of cost-effectiveness of manual and mechanical debarking methods, assuming an hourly contracting rate of 50€/h

Description	Value	Description	Value
Hourly rate (€/h)	50	Manual labor cost (€/kg)	5
Manual debarking time (min/kg)	6	Mechanical labor cost (€/kg)	0,02083333
Mechanical debarking time (min/kg)	0,025	Cost savings per kg (€)	4,97916667
Mechanical upfront investment (€)	10000	**Break-even weight (kg)**	2008,3682

18.49 kg of poplar trunks were processed, which yielded 14.9 kg of trunk biomass and 3.47 kg of bark. The trunks were partially dried over 3 weeks to prevent the formation of mould over the transport period. The drying process reduced the weight by 35%, leaving 9.5 kg of wood and 2.53 kg of bark, which were sent to CETIM. The debarkation efficiency was 100%, as purity of the wood was prioritised over the minimisation of material loss while establishing the process.

4. DRYING AND SHREDDING OF BIOMASS

Within the framework of the pHYBi project, the poplar biomass obtained from phytomanagement strategies was subjected to a physical pre-treatment aimed at standardizing its properties prior to fractionation and valorisation stages. This preliminary conditioning is essential to ensure experimental reproducibility, material homogeneity, and the efficiency of subsequent processing steps.

At CETIM, different types of poplar biomass were received from UMLP: debarked poplar trunk (debarked by PHY, figure 1) and poplar branches (figure 2). The diversity of plant fractions (trunk wood and branches) required a consistent and standardised pre-treatment protocol to minimise variability in subsequent processing steps.



Figure 1. Debarked poplar trunk



Figure 2. Poplar branches

4.1. BIOMASS DRYING

The collected biomass exhibited a significant moisture content, both surface-bound and structurally embedded within the lignocellulosic matrix. In order to remove free and interstitial water and to prevent potential physicochemical or microbiological alterations during storage, the material was dried in an ARGOLAB TCF400 oven (figure 3) under controlled conditions until constant weight was achieved. After several temperature and time tests, 60ºC and 24 h were chosen as the optimal drying conditions.



Figure 3. ARGOLAB TCF400 oven

Drying to constant mass enabled all subsequent operations to be conducted on a dry-weight basis, thereby reducing variability associated with moisture content and ensuring greater accuracy in mass balances and yield calculations. Furthermore, moisture reduction enhances milling efficiency and promotes a more uniform particle size distribution by decreasing material plasticity and agglomeration tendencies.

4.2. PARTICLE SIZE REDUCTION BY SHREDDING

Following drying, the poplar biomass was subjected to particle size reduction using a Restch SM200 knife mill (figure 5). Prior to this, the biomass was cut using a power saw (figure 4) to reduce it to a size of between 3 and 4 centimetres, which proved to be the most suitable for feeding the sample into the knife mill, as shown in figures 6 and 7. The initial idea was to achieve this particle size of 3-4 centimetres of length, as this was considered sufficient for subsequent characterisation. However, we considered this size to be too large for the organosolv process to be effective, as the literature contains several cases of organosolv treatment of poplar in which the biomass is ground to a particle size of between 0.5 and 1 mm[1,2].

[1] Kim, D.-E. and Pan, X. (2010). Preliminary study on converting hybrid poplar to high-value chemicals and lignin using organosolv ethanol process. *Industrial & Engineering Chemistry Research, 49*(23), 12156–12163.

[2] Sun, Q., Khunsupat, R., Akato, K., Tao, J., Labbé, N., Gallego, N. C., Bozell, J. J., Rials, T. G., Tuskan, G. A., Tschaplinski, T. J., Naskar, A. K., Pu, Y., & Ragauskas, A. J. (2016). A study of poplar organosolv lignin after melt rheology treatment as carbon fiber precursors. *Green Chemistry, 18*(18), 5015–5024.



Figure 5. Power saw



Figure 4. Retsch SM200 knife mill



Figure 6. Poplar trunk after power saw



Figure 7. Poplar branches after power saw

The milling process was carried out in two sequential stages in order to obtain a controlled granulometry suitable for downstream fractionation treatments. In the first stage, a coarse grinding was performed to achieve an approximate particle size of 4 mm. This step allowed material homogenisation, volume reduction, and facilitated handling and intermediate storage. Additionally, the initial size reduction helped minimise the inherent structural heterogeneity of woody biomass, which typically consists of fibres, vascular tissues, and bark fractions with different mechanical resistances.

Subsequently, a second milling stage was conducted to obtain a finer particle size of approximately 0.5 mm. Further size reduction increases the specific surface area of the material, which is critical for improving reagent accessibility during the organosolv fractionation processes envisaged in the project. Moreover, a finer granulometry enhances sample homogeneity in compositional analyses and valorisation assays,

thereby contributing to improved reproducibility and comparability of experimental results. The result of the particle size reduction can be observed in the figures below.



Figure 8. Poplar trunk dried and shredded



Figure 9. Poplar branches dried and shredded

A third milling stage was also carried out in an attempt to obtain the smallest possible particle size. As shown in the figure 10, a particle size of 125 µm was obtained. However, more than half of the sample was lost during sieving, so this final milling stage was discarded. The particle size obtained of 0.5 mm was considered adequate to ensure homogeneity and reagent accessibility for organosolv treatment and compositional analyses in the next stages of the project.



Figure 10. Feedstock after third milling stage

5. DISCUSSION OF RESULTS

The manual and mechanical debarkation methods were evaluated and key sample weights for the economic viability of each process were estimated based on the process duration and hourly costs. The scope of the pHYBi project as a research endeavour likely will not exceed this key sample weight. Nonetheless, the mechanical process should be included in the techno-economical and life-cycle assessment of the valorisation process since it certainly would be used for the industrial-scale process that pHYBi envisions.

Regarding drying and shredding operations, various parameters were assessed throughout the process, such as initial and final moisture content, particle size and material losses. The results obtained are shown in the following tables:

Table 2. Parameters of drying and shredding operations

Fraction	Initial moisture	Final moisture	Particle size
Trunk	36,24%	3,04 %	0,5 mm
Branches	39,84%	5,24 %	0,5 mm

Table 3. Mass balance during processing steps of poplar trunk and branches

Fraction	Initial biomass	Debarked wood	Bark	After drying (PHY)	After drying (CETIM)	After milling (CETIM)
Trunk	18,49 kg	14,9 kg	3,47 kg	9,5 kg	-	8,85 kg
Branches	30,45 kg	-	-	-	18,32 kg	17,11 kg

In light of the results, we can highlight that the biomass moisture content has been effectively reduced, yielding an optimal particle size for the subsequent stages of the project. Furthermore, material loss during the process has not been significant, so it can be concluded that the feedstock pre-treatment has been both effective and efficient.

6. CONCLUSION

The manual debarking process for large trunks was applied and yielded satisfactory results. This method will continue to be used for further rounds of processing.

The physical pre-treatment applied to the different lignocellulosic biomasses within the framework of the project proved to be an essential step to ensure material standardisation prior to fractionation and valorisation processes. Drying the biomass at 60 °C for 24 hours until constant weight efficiently removed moisture, enabling processing on a dry-weight basis while preventing degradation during storage and improving milling efficiency.

Sequential shredding produced a controlled and homogeneous particle size. The first stage (4 mm) facilitated handling and homogenisation, while the second stage (0.5 mm) increased surface area and improved reagent accessibility for organosolv fractionation and analytical reproducibility. A third milling stage (125 µm) was tested but discarded due to significant material losses during sieving.

Overall, the drying and two-stage shredding protocol provided suitably conditioned poplar biomass for subsequent processing.