Chemical and Thermogravimetric Characterization of Chestnut Tree Biomass Waste: Towards Sustainable Resource Utilization in the Tannin Industry

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ABSTRACT

Sweet chestnut (Castanea sativa Mill.) is a key European species, with Italy playing a crucial role in its forest cover and timber production. Chestnut wood processing generates significant residues, with approximately 40% waste at the sawmill stage alone. A major portion of this waste, including that from fruit tree plantations, is used in the tannin extraction industry, exemplifying a circular economy model. This study analyzed exhausted chestnut wood from the tannin industry, virgin wood (prior to tannin extraction), detannized wood (after tannin removal), and chestnut bark, which is separated before the tannin extraction process. The study assessed both the chemical composition and extractives content of the samples. FTIR analysis revealed structural modifications, with key spectral shifts (1640, 1420, 1240, and 1120 cm^{-1}) indicating enhanced aromatic conjugation after extractive removal. TGA analysis showed that detannized wood degraded faster at high temperatures, suggesting better combustibility and biofuel potential, while lignin-rich residues demonstrated greater thermal stability, making them suitable for insulation and biocomposite applications. These findings support optimizing waste utilization, reducing environmental impact, and enhancing resource efficiency. By promoting innovative uses for tannin industry byproducts, this research contributes to sustainability and strengthens circular economy strategies within the forest-wood sector.

Keywords: Exhausted chestnut wood, detannized wood, chestnut bark, tannins, extractives, HPLC, HPSEC, FTIR, TGA

1. INTRODUCTION

Sweet chestnut *(Castanea sativa* Mill.) is the only native species of the genus in Europe. The main chestnut refugia are thought to be the Transcaucasian region, northwestern Anatolia, the hinterland of the Tyrrhenian coast from Liguria to Lazio along the Apennine range, the region around Lago di Monticchio (Monte Vulture) in southern Italy, the Cantabrian coast on the Iberian Peninsula, and probably also the Greek Peninsula (Peloponnese and Thessaly) and northeastern Italy (Colli Euganei, Monti Berici, Emilia-Romagna) (Krebs et al., 2004). In the context of the forestry industry in Italy, the sweet chestnut plays an important role, contributing significantly to the country's forest cover, encompassing approximately 7.5% of the total forest area (Manetti et al. 2017; Marini et al., 2020). Currently, forest chestnut trees primarily serve the purpose of timber and load-bearing structures (Marini et al., 2020). However, wood processing results in significant amounts of residue (Tamantini et al., 2021; Carbone et al. 2023), which is estimated to be approximately 40% by volume from the round log, considering

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only the initial step of the forest-wood chain. Wood residues originating from sawmills, forest harvesting and the cultivation of chestnut fruit tree plantations are commonly managed by direct combustion methods (pellet, biomass for energy). However, a significant portion of these residues are directed for tannin extraction industries located in northern Italy (San Michele Mondovi - Piedmont) and central Italy (Radicofani - Tuscany).

Previous studies have extensively characterized chestnut tannins, recognizing their applicability in diverse sectors such as winemaking (Canas et al., 2016), pharmaceuticals and cosmetics due to their antioxidant and astringent properties (Aires et al., 2016), traditional leather tanning (Vázquez et al., 2008), and natural adhesives (Spina et al., 2013). Owing to their antioxidant and astringent features, tannins are used in both pharmacological products and the cosmetics industry (Aires et al. 2016). Their traditional and historic use is related to leather processing, facilitating the transformation of hides and skins into leather and acting as retaining agents (Korel and Balaban, 2009). Tannins are added to animal feed because they can act against pathogens (Krueger et al. 2010). Tannins also have environmental applications because they can absorb heavy metals from aqueous solutions (Akunwa et al. 2014, Fan et al. 2014). Finally, adhesives must be considered, and tannins continue to remain a meaningful ingredient in natural adhesives and coatings (Spina et al. 2013, Aires et al. 2016, Santos et al. 2017, Marini et al., 2021). All these applications indicate that tannins from natural origins represent an important resource for achieving sustainable development and a circular economy (Zikeli et al. 2018, 2019), and the reuse of forest residues, crops and industrial wood waste represents a major achievement for combating environmental issues (Aires et al. 2016, Ham et al. 2015, Kim et al. 2015, Vázquez et al. 2008). After extracting tannins from chestnut wood, the tannin industry produces a high amount of exhausted lignocellulosic biomass (thousands of tons), which needs to be considered a potential resource for high added value chains (Marini et al. 2002). Recently, there has been a growing trend toward identifying high-value applications for waste resources (Jusic et al. 2021., Zikeli et al. 2019), especially considering the potential fractionation of residual lignocellulosic biomass, principally comprising lignin and various carbohydrates, within a biorefinery concept (Tamantini et al., 2021). Each type of transformation from a biorefinery perspective can be pursued only from the perspective of the original biomass input. The chemical composition of the lignocellulosic input can be very different depending on the provenance, age, and storage conditions (Tamantini et al., 2021). Despite extensive research on tannins themselves, the chemical and structural properties of the exhausted biomass from tannin extraction remain poorly characterized, leaving a significant knowledge gap regarding its potential for higher-value industrial application. Every year, thousands of tons of these exhausted biomass, mainly composed of lignin, cellulose, hemicellulose, and extractives, remain underutilized or are directed towards low-value applications. Understanding the composition and behavior of these residues is crucial in the context of sustainable resource utilization and the development of biorefinery strategies. In order to reach such goal, this gap, this study conducts a comprehensive chemical characterization of chestnut wood residues before and after tannin extraction. The chemical composition characterization is crucial to evaluate further fractionation processes such as organosolv for the recovery of biopolymers, cellulose and lignin. In particular, the determination of extractive content will be valuable to evaluate the recovery of such components for further high-value applications such as antioxidant and antimicrobial

By analysing the content of cellulose, hemicellulose, lignin, and extractives, aims to assess the compositional changes induced by the extraction process. Structural modifications in the biomass are examined using Fourier transform infrared (FTIR) spectroscopy, providing insights into alterations in chemical bonding and functional groups. Additionally, thermogravimetric analysis (TGA) is employed to evaluate the thermal stability and degradation behavior of both raw and exhausted residues, determining their suitability for bio-based material applications and energy production. Thermal stability analysis would help the evaluation in pollutant adsorbent both for air and water area of applications and, in the preparation of composites (Santos et al., 2022).

Particular attention is given to chestnut bark, as it represents a significant portion of the biomass waste and has demonstrated potential for biorefinery applications. By providing a detailed understanding of these residues, this research seeks to propose innovative ways to optimize their utilization, supporting the transition toward a more sustainable and circular forest-wood industry.

2. MATERIALS AND METHODS

2.1 MATERIALS

The wood material used was provided by Nuova Rivart (Srl), the tannin industry in Radicofani (SI, Italy), and consisted of three different batches of wood waste. The initial batch was virgin wood waste (VW), indicating its intact state prior to any alterations or treatments. The second batch comprised chestnut wood bark (B), also classified as virgin material, since it is removed before starting the industrial process of tannin extraction. The subsequent batch was a detannized wood waste (DT), the residual material after the tannin extraction process at the industrial facility. Notably, the extraction process for hydrolysable tannins from chestnut trees involves the use of a water-based methodology. The exhausted biomass waste obtained after extraction represents a heterogeneous mixture of lignocellulosic material, making it impossible to distinguish between heartwood and sapwood or to determine whether it originated from a single trunk or multiple sources.

(a) (b) (c)

Figure 1. (a) Exhausted chestnut wood; (d) exhausted chestnut wood powder; (c) chestnut wood extractives

The subsequent batch was a detannized wood waste (DT), the residual material after the tannin extraction process at the industrial facility. Notably, the extraction process for hydrolysable tannins from chestnut trees involves the use of a water-based methodology. The exhausted biomass waste obtained after extraction represents a heterogeneous mixture of lignocellulosic material, making it impossible to distinguish between heartwood and sapwood or to determine whether it originated from a single trunk or multiple sources. However, it is important to consider that chestnut wood possesses only a very thin sapwood layer; therefore, it can be reasonably assumed that the analyzed feedstock consists predominantly of heartwood. To ensure reproducibility and minimize the effect of a different moisture content in the investigated material, all wood samples Virgin (VW), detannized (DT) and the bark (B) were uniformly dried at a room temperature (approximately 20–25°C) and humidity which is about 60%, until reaching equilibrium moisture content at environmental condition. After reaching consistent moisture levels (~10% moisture content), the samples were milled to pass through a 35-mesh sieve using an IKA M10.1 cutting mill (IKA®-Werke GmbH & Co. KG, Staufen, Germany). The final product obtained was wood powder with an average particle size of 0.25 mm (Fig. 1).

2.2. METHODS

2.2.1. Soxhlet extraction with acetone

The milled wood was Soxhlet-extracted for 6 h with acetone (Sigma–Aldrich, Milan, Italy) in a round-bottom flask, and the experiment was repeated three (3) times. After extraction, the extract was concentrated with a rotatory evaporator and eventually freeze-dried. The extraction yield was calculated according to the equation below:

$$\text{Extraction yield} = \frac{\textit{mass of the solid residue}}{\textit{dry mass of sawdust}} \text{ x } 100 \qquad (1)$$

Acetone was selected as the extraction solvent due to its intermediate polarity, which enables the efficient extraction of a broad spectrum of wood extractives (Omar et al., 2022). It is particularly effective in extracting phenols, fats, and fatty acids from wood (Demirbas, 1991) and is widely used for sample preparation prior to the fractionation of lignocellulosic biomass into its fundamental components (Zikeli et al., 2019). Moreover, the low volatility allows an easier removal with respect to other green solvents such has 2-methyl-tetrahydrofuran, cyclopentyl methyl ether and cyrene. Also, it is a cheap solvent suitable for scaling up considering the high amount of biomass that can be valorized. Other solvents, known for high extraction capacity are toxic such as benzene, chloroform and dichloromethane, this last aspect makes acetone a safer and more environmentally favorable option (Demirbas, 1991; Solikhin et al., 2021).

2.2.2. High-performance liquid chromatography (HPLC) and high-performance size exclusion chromatography (HPSEC)

The concentrations of both C5 and C6 carbohydrate monomers were detected via a Dionex Ultimate 3000 HPLC (Thermo Scientific, Sunnyvale, CA, USA) instrument equipped with a Bio-Rad Aminex HPX-87H column (Bio-Rad, CA, USA) and a RefractoMax 520 RI detector (Thermo Scientific, Waltham, MA, USA). The analysis conditions were set as follows: 50°C for both the column oven and the detector, 0.01 N H_2SO_4 for the mobile phase, and 0.6 mL/min for the eluent flow. The concentration of polymeric sugars was calculated using an anhydrous correction of 0.88 and 0.90 for the C5 and C6 carbohydrates, respectively (Aiello et al., 2021). Since the aforementioned column does not separate a mixture containing xylose, mannose and galactose, instead giving a combined peak for all those sugars, samples were also run using a Thermo-Fisher HyperREZ Pb2+ Column to allow for the discrimination of the concentrations of xylose, mannose and galactose using the same previously described equipment. The analyses were run via an internal method optimized from the column factory's standard method: temperatures were set to 70°C for the column oven and 55°C for the RI detector. Deionized water (15 MΩ) was used as the mobile phase. The eluent flow rate was set to 0.6 mL/min.

The molecular weight (M_w), number-average (M_n) and polydispersity index (M_w/M_n) of the isolated extractives were analyzed by alkaline high-performance size exclusion chromatography (HPSEC), an Agilent 1100 HPLC system equipped with a temperated column compartment and a diode array detector using Agilent ChemStation (version B.03.02) for operation (Agilent Technologies Inc., Santa Clara, CA, USA). Molar mass analysis was performed using a PSS MCX column with a 5 µm particle size and 1000 Å pore size (PSS Polymer Standard Services, Mainz, Germany) which was maintained at 40°C. The mobile phase was a 10 mM NaOH solution with the addition of 20 mM $NaNO_3$ as a neutral salt. The flow rate was set to 0.5 mL/min, and the UV signal at 280 nm was used for molar mass determination. For calibration, polystyrene sulfonate standards were used with the following molar masses at peak maximum: 65,400, 33,500, 15,800, 6430, 1670, 891, and 208 Da. Weight- and number-average molar masses were calculated via the GPC add-on (version B.01.02) of Agilent ChemStation software.

2.2.3. Fourier transform infrared (FTIR) spectroscopy

Samples of wood powder mixed with potassium bromide (KBr) pellets (Ø = 7 mm) were prepared with a concentration of 2 wt.% using Specac mini pellets pressed at 2 bar for 5 min (Specac Inc., Orpington, UK). FTIR spectra were recorded in absorbance mode in the range of 4000–400 cm^{-1} with a FTIR-4100 Fourier transform infrared spectrometer (Jasco Corp., Easton, MD, USA). All the FTIR spectra were smoothed and baseline corrected. Although infrared scanning was performed in the region between 4000 and 400 cm^{-1}, data evaluation was limited to the fingerprint region (1800–900 cm^{-1}), in which most of the variations in infrared absorption occurred.

2.2.4. Thermogravimetric analysis (TGA) and calorific value

A thermogravimetric analyzer TGA/DSC 3+ STARe System (Mettler Toledo, Columbus, OH, USA) was used to study pyrolysis behavior. The samples were placed in a 150 µL Al2O3 pan and heated from 25°C to 900°C at a heating rate of 10°C/min under an inert atmosphere with a nitrogen flow rate of 50 mL/min. The weight loss (TG) curves and their respective first-order derivatives (differential thermogravimetric (DTG)) were recorded over time and at different temperatures. The calorific value of the samples was measured according to UNI EN ISO 18125:2018 via an isoperibolic calorimeter (AC-350, Leco Corporation), where 1.0 g of wood powder was placed in a sealed "bomb", and the fuel was first combusted with oxygen, under pressure. Then, the heat of the subsequent reaction was measured from the temperature rise of a surrounding water bath. At the end of the 10-minute process, the caloric value (MJ/kg) of each sample was determined.

3. RESULTS

3.1. EXTRACTIVE CONTENT AND CHARACTERIZATION

Extractives are chemical compounds found in lignocellulosic sources; these substances usually include phenols, resin, waxes, and oil residues (Solikhin et al. 2021). Water and organic solvents, such as ethanol, benzene, acetone, toluene, ether, and hexane, can be employed for extractive removal, and different solvents can impact the amount and type of extractives produced (Solikhin et al. 2021). There is no solvent able to affect the complete removal of wood extractives. In our investigation it was decided to use only one solvent acetone, because water was already used during the process of tannin extraction at industrial scale. Acetone offers several advantages as a solvent, primarily due to its relatively low toxicity, which classifies it among the most preferred solvents with minimal health hazards. It is widely utilized in processing lignocellulosic biomass into high-value products, including aerogels (Bujanovic 2018). Acetone effectively extracts phenolic compounds and is frequently employed in fundamental studies for determining the chemical composition of biomass. Additionally, it plays a role in organosolv processes, aiding in lignocellulosic biomass fractionation by facilitating lignin separation and improving cellulose accessibility for further processing (Tanis et al., 2023; Pang et al., 2021). Among organic solvents, acetone, along with ethanol, is expected to be increasingly utilized in second-generation biorefineries, particularly in pretreatment processes (Bujanovic, 2018). Its solubility parameter aligns well with lignin, making it an excellent choice for fractionation and purification studies, as well as for producing lignin fractions of varying sizes. Recently, ionic liquids and deep eutectic solvents (DES) have been explored as environmentally friendly alternatives, demonstrating potential in the removal of wood extractives (Solikhin et al., 2021; TAPPI, 2007). Ethanol, a renewable solvent, is also widely used due to its low toxicity and broad applicability (Solikhin et al., 2021; TAPPI, 2007). From the virgin wood sample (VW) extractives, 7.17% were extracted, and the provenance of the chestnut wood closely resembled that studied by Zikeli et al. (2019), primarily in Central Italy, including the Latium and Tuscany regions. Consequently, the quantity of extractives obtained aligns closely with the findings reported there (6.6%), as our study applied identical extraction procedures and conditions. Vidrich (1988.) - reported similar numbers, 6.8%, indicating that the contents of tannins and extractives (not only tannins) in the chestnut tree reach maximum values at approximately 37 years, whereas for the bark, the maximum values occur at the age of 10 years. In exhausted wood (DT) after acetone-based extraction, extractives are still present in the residual biomass (4.02%). The industrial process of tannin extraction typically involves the use of hot water and pressure to extract tannins from lignocellulosic material. This process indeed leaves behind a significant amount of compounds, particularly phenolic compounds, in the residual biomass (Giovando et al., 2019). These phenolic compounds are often regarded as valuable secondary products or coproducts of the tannin extraction process, and some efforts have been made to determine the potential utility of these residual compounds because their antioxidant and antimicrobial properties are still present (Panzella, 2019). In our investigation, the combination of different solvents can be highly beneficial for the more successful removal of wood extractives. The bark of chestnut wood presented the lowest percentage of extractives (2.5%), but despite this, bark extracts are known to contain significant amounts of phenolic compounds, such as castalin, vescalin, ellagic acid and gallic acid which are quite important for high added value chain (Comandini et al. 2014). These results contrast sharply with the findings of Vidrich (1988), who reported that chestnut bark contains hydrolysable tannins and phlobaphenes of around 13%.

The molar mass averages determined by HPSEC are reported in Table 1. All extracts consisted of a heterogeneous mixture of compounds with different fractions. The molar mass averages differ greatly between the samples, and the dispersity (Đ) is rather high. The highest M_w was obtained for a detannized wood sample (DT), with a dispersity of 73.15. Few articles have reported HPSEC characterization of extractives, and the extraction methods used are not the same as those used in this study; therefore, comparisons with the literature must be performed carefully (Sillero et al., 2019). Vidrich (1988) reported that tannins possess a considerable molar mass, with chestnut tannins reaching up to 1550 μm. Differences between the molar masses of the bark extractives reported in the literature and our results exist. In the study conducted by Sillero et al. (2019), various bark extracts from diverse tree species were examined. The highest average molecular weight, 57387 g/mol, was observed in sweet chestnut bark, with a dispersity of 27.99, which was significantly greater than that in our study. The inconsistencies in molar mass across the extracts can be attributed to the heterogeneous nature of extractives, the influence of the extraction method, chemical modifications during extraction, and the potential degradation or polymerization of certain components. The detannized sample (DT), with its exceptionally high dispersity, suggests the presence of a mixture of molecules with great differences in molar mass. When comparing the HPSEC elugrams of the extractives from virgin wood (VW), from detannized wood (DT), and from industrial tannin (IT), respectively, it is evident that the fraction of hydrolyzable tannins has already been removed during industrial tannin extraction. Thus, in DT, still present high-molar-mass condensed tannins were eventually extracted using acetone beside low-molar-mass compounds soluble in acetone, which could derive from lignin. The removal of hydrolyzable tannins through industrial hot water extraction likely increases lignin susceptibility, as the extraction process exposes the cell wall to solvents. This enhanced exposure can influence the wood's surface characteristics, potentially affecting its roughness (Chavenetidou & Kamperidou, 2024). Conversely, the industrial tannin extract exhibited the lowest Mw and dispersity, likely due to simply hot-water extraction and eventual purification processes. These variations highlight the influence of extraction conditions and chemical composition on molecular weight distribution in chestnut wood extractives.

Table 1. Average percentage of extractive content in the three samples and molar mass averages (weight-average molar mass M_w, number average molar mass M_n) of the respective extractives obtained by acetone solution.

Sample	**No. of samples (30 g per sample)**	**Mean of extractives (%)**	**St. Dev**	**Extractives M_w (g/mol)**	**Extractives M_n (g/mol)**	**Đ**
VW	3	7.17	0.49	15400	1014	15.19
DT	3	4.02	1.54	95100	1300	73.15
B	3	2.5	0.058	18340	603	30.41
Industrial tannin				2850	1183	2.41

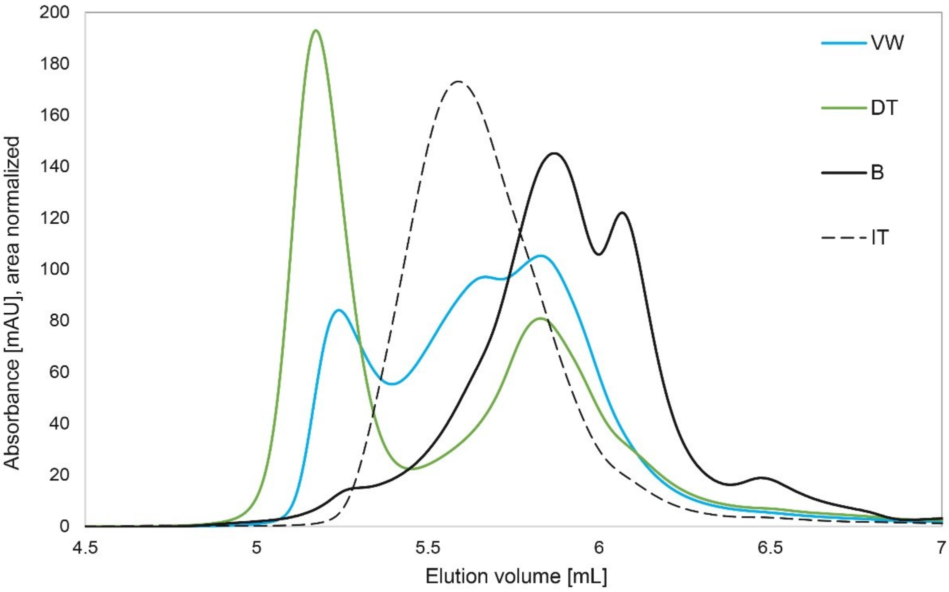

Figure 2. HPSEC elugrams of the extractives fractions from chestnut virgin wood (VW), detannized chestnut wood (DT), chestnut bark (B), and an industrial chestnut tannin sample (IT).

Noteworthy, the tannin content extracted with acetone is lower compared to the comparable feedstock of exhausted chestnut examined by Panzanella et al (2019). However, in the case of the cited authors, it should be noted that the further extraction of the detannized biomass was carried out using acetone and followed by acetic acid. Moreover, it should be considered that the molar mass (Mw and Mn) increases in the case of DT with a high dispersity value, which likely indicates a greater ability of acetone to interact with the constituent components of the cell wall.

The major chemical component of living trees is water, but on a dry weight basis, all wood cell walls consist mainly of sugar-based polymers (carbohydrates, 65–75%), which are combined with lignin (18–35%) (Rowell R.M., 2005). The results from the main chemical composition, including the contents of cellulose, hemicellulose, and lignin in the three samples after the extractives were removed, are presented in Table 2.

Table 2. Chemical composition of the samples

Sample	No. of samples	Average of hemicellulose (%)	Xylose[1] (%)	Galactose[1] (%)	Other carbohydrates (%)	Average of cellulose (%)	Average of lignin (%)	Not identified components[2] (%)
VW	3	13.90	96.94	3.07	<1	36.66	31.89	17.56
DT	3	13.40	93.69	5.31	<1	37.78	29.38	19.38
B	3	9.58	96.67	2.33	<1	27.79	33.63	29.10
St. Dev		1.36				2.03	0.24	0.035

[1] *The percentage of xylose and galactose are per mass of hemicellulose (example: out of 100 g of hemicellulose, 74.43 g are xylose)*

[2] *Acetyls, pectin, extractives, ashes, and sugar degradation products*

The lignin content across all three samples was relatively consistent, with a slight increase observed in the bark samples (33.63%). Similarly, the cellulose content remained uniform between the virgin (36.66%) and detannized (37.78%) samples, although it was slightly lower in the bark (27.79%). The hemicellulose content showed similar trends, with the lowest concentration in the bark (9.58%). Unidentified components of the samples are the remains of the extractives, acetals, pectins, ashes and products of sugar degradation. As expected, the tannin extraction process had a slight effect on the percentage of cell wall components (lignin/cellulose), except for a slight decrease in the soluble fraction of lignin as was mentioned above.

3.2. FOURIER TRANSFORM INFRARED SPECTROMETER

The FTIR spectra of virgin wood waste (named after VW_be) and the samples after acetone extraction from virgin wood waste (VW_noextr), chestnut wood bark (B_noextr), detannized wood (DT_noextr) and chestnut tannins are shown in Figure 3. The peak assignments were conducted according to the literature and are summarized in Table 2. In the “fingerprint” region, a high level of complexity is observed, characterized by numerous bands attributed to the primary components of wood: many sharp and discrete absorption bands arise owing to diverse functional groups present in wood constituents. Notably, specific bands at 1599, 1510 and 1240 cm^{-1} correspond to the characteristic bending or stretching of different groups of lignin. Similarly, the bands at 1461, 1423, 1378, 1154 and 1030 cm^{-1} are associated with the characteristic bending or stretching vibrations of different groups found in both lignin and cellulose. Furthermore, the bands at 1739 cm^{-1} are linked to characteristic bending or stretching vibrations of the different groups of cellulose. The main characteristic peaks, which are the stretching vibrations of C=O aromatic and C-O, typically occur at approximately 1640, 1510 and 1030 cm^{-1}. The O-H, C-H and C=O stretching modes above 1600 cm^{-1} along with the aromatic skeletal vibration band at approximately 1510 cm^{-1} are pure, whereas the 1600 cm^{-1} aromatic skeletal vibration band represents a signal that has been broadened by the C=O stretching mode. Other bands in the fingerprint regions below 1460 cm^{-1} are complex, stemming from various vibration modes in carbohydrates and lignin. Importantly, most of the bands in the fingerprint region derive contributions from all the wood constituents, demanding careful interpretation of the IR data. Nonetheless, variations in positions and relative intensities are observed among the four types of wood samples. The band intensity at 1739 cm^{-1} was greater in DT_noextr than in B_noextr and VW_noextr. The highest value for this peak was observed for the tannins. This band is attributed to the stretching of the free carbonyl groups. A high signal intensity in the 1700–1760 cm^{-1} range often indicates the presence of hemicellulose in milled wood lignin. Phenolic acid esters, particularly those found in grass lignin, might also influence the signal within this range. The bands at approximately 1315 cm^{-1} are visible due to the condensation of S and G units and to the bending of O–H bonds in polysaccharides, including cellulose and hemicellulose. The spectra of the fingerprint regions of the three wood samples are rather similar, as 11 out of the 15 discernible bands are shared by the samples. B_noextr and DT_noextr reflect strong and similar absorbances in the regions of bond vibrations typical of cellulose and lignin. Compared with the B_noextr and DT_noext samples, the VW_noextr sample had a lower absorbance for every peak except for the band at 1462 cm^{-1}. The B_noextr spectra also exhibits more bands than the other two samples do in the region between 1555 and 1521 cm^{-1}.

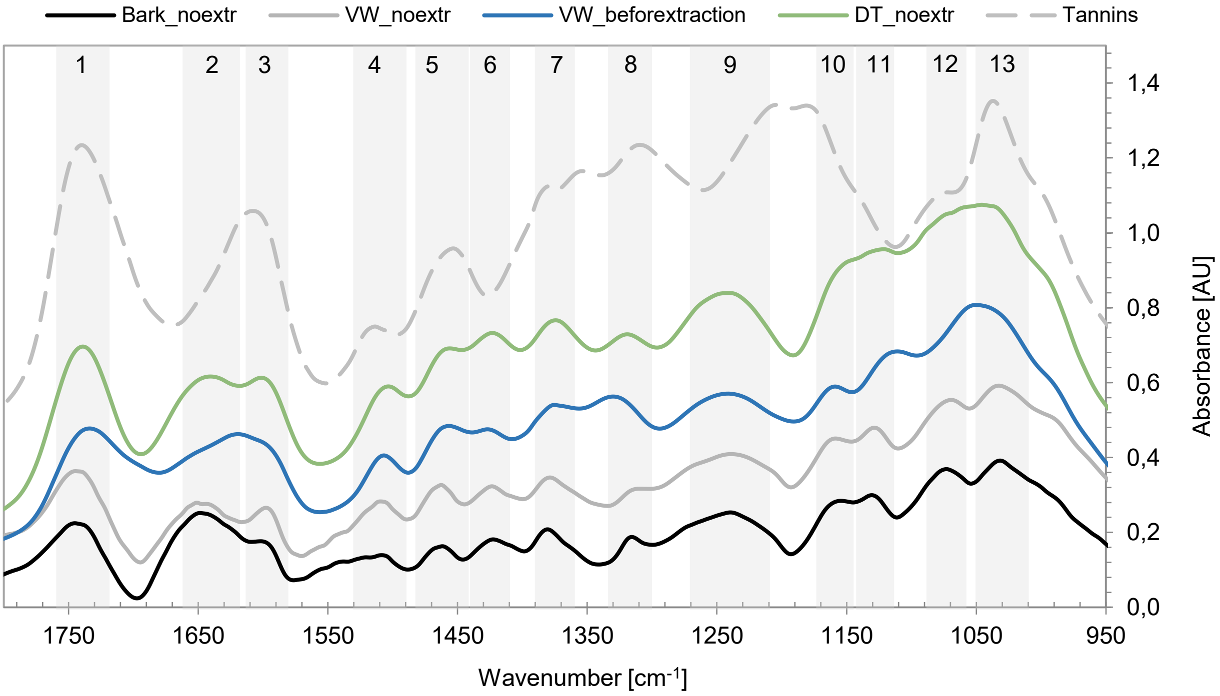

Figure 3. FTIR spectra of the samples and comparison with an industrial tannin spectrum

Considering the VWs before and after extraction, new bands, such as those at 1650, 1640, 1597, 1310, 1069 and 1032 cm^{-1}, appeared after extraction, whereas other bands, such as those at 1618, 1329, 1110 and 1050 cm^{-1} disappeared. Tannins can be detected in the VW_be spectra by observing the characteristic absorption bands at 1618 cm^{-1} and 1050 cm^{-1}. Additionally, the band at 1329 cm^{-1} represents the vibration of C-O-C stretching and OH deformation before extraction, indicating the presence of hydrolyzable tannins, namely, ellagitannins. In addition, in the VW_be spectra, the band at 1160 cm^{-1} coupled with the band at 1110 cm^{-1} represents the condensed tannin group. A comparison of the tannin spectra with the three samples' spectra revealed that the wood removal of extractives (i.e. tannins) resulted in the onset of bands at 1640, 1420, 1240, and 1120 cm^{-1}. All these bands are related to the aromatic portion of the wood, indicating that better conjugation of the aromatic ring is possible due to the removal of extractives. The removal of tannins also resulted in the disappearance of the band at 1179 cm^{-1}. Both the comparison between VW before and after extraction and the comparison between tannins and B, DT, and VW seem to show that acetone can enhance the presence of lignin, i.e. aromatic compounds and cellulose; as a result, the lignin-associated bands are clearly visible. The FTIR results indicate that detannized wood (DT) exhibits a higher exposure of functional groups, including carbonyl and hydroxyl groups. These findings suggest that DT biomass may have improved potential to produce bio-based adhesives, biopolymers, and coatings, where carbonyl functionalities contribute to better cross-linking and adhesion properties, and a more accessible lignin, which is one of the building blocks of the future. Additionally, the spectral changes in the lignin-associated bands suggest that detannized biomass could serve as a reinforcement material in composite production due to enhanced interactions between lignin and synthetic resins. The observed presence of exposed cellulose and hemicellulose structures in DT and B samples suggests potential use in biofuel and biochemical production, as these components can be more easily hydrolyzed into fermentable sugars. The increase in lignin-related bands also supports the idea that these residues could be valorized for high-value lignin-derived compounds, such as bio-based phenolic resins or antioxidant-rich extracts for industrial applications.

Table 2. Main functional groups and peaks of the three samples and tannins

Wavenumber (cm^{-1})					**Base group and vibration mode**
VW_noextr	**B_noextr**	**DT_noextr**	**Tannins**	**VW_be**	
1745	1745	1739	1739	1733	C=O stretching in unconjugated ketone, carbonyl and aliphatic groups in xylan (hemicellulose)[a]
1641	1648	1641	-	-	C=O stretching conjugated to aromatic ring[b]
1597	1599	1601	1608	-	C=O stretching conjugated to aromatic ring[c]
1510	1508	1503	1513	1507	aromatic skeletal vibration plus C=O stretching[c]
1462	1461	1456	1453	1456	C-H deformation stretching in lignin and carbohydrates, C-H deformation (methyl and methylene)[c, d]
1423	1422	1423	-	1427	aromatic skeletal combined with C-H in-plane deforming and stretching[e]
1378	1380	1374	1379	1375	C-H deformation in cellulose and hemicellulose[f]
1310	1315	-	1309	-	condensation of G and S units, S unit and CH2 bending stretching, O-H bending[c]
1239	1240	1241	-	1240	syringyl ring and C-O stretch in lignin and xylan[g]
1158	1154	1151	-	1159	C-O-C vibration in cellulose and hemicellulose[g]
1128	1131	1121	-	-	aromatic skeletal and C-O stretch[e]
1069	1073	-	1077		C-O deformation in secondary alcohol and aliphatic ethers[e]
1032	1032	1028	1037		C-O deformation in primary alcohols, plus C=O stretch unconj., plus aromatic C-H in-plane deformation[e]

[a] (Sarken, Chang&Ericsson 1967); [b] (Adler 1977, Fengel&Shao 1985); [c] (Chen&Ferrari 2010); [d] (Popescu 2007), [e] (Faix 1992), [f] (Yang 2015), [g] (Özgençet al. 2017)

3.3. THERMOGRAVIMETRIC ANALYSIS AND CALORIFIC VALUE

The influence of various wood extractives on the thermal stability of wood has been investigated. Mészáros et al. (2007) reported significant differences between extracted and non-extracted wood samples. Figure 3 shows the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves for the three types of wood samples; this influence is particularly noticeable. The presence of extractives in the virgin wood and chestnut bark samples affects the decomposition behavior of the biomass. In contrast, the effect of extractives is less visible in the detannized wood sample. The main thermal degradation intervals can be summarized by examining the percentage of weight loss as a function of temperature, as shown in Fig. 4. The residual water loss was observed from 25°C to 100°C, as expected. The second thermal degradation interval takes place at approximately 275°C, and a shoulder can be observed in all the samples, although more clearly in the DT. At approximately 350°C, the main degradation of cellulose occurs, and a prominent peak can be observed, corresponding to the maximum decomposition rate. According to Kim et al. (2006) and Poletto et al. (2012), the depolymerization of hemicellulose occurs between 180°C and 350°C. The random cleavage of glycosdic linkages increases the sensitivity of hemicellulose to thermal degradation. Cellulose degradation occurs mainly between 275 and 350°C, and lignin degradation starts at 250°C, with partial overlap with cellulose and usually ends at 500°C. Lignin is known to be the most difficult to decompose, and its decomposition occurs slowly over the whole temperature range from ambient temperature to 900 °C (Yang et al. 2007). We observed that in the detanninized sample, the rate of decomposition was higher at approximately 375°C, indicating that it burned more rapidly. In contrast, as expected, the untreated bark burns less efficiently due to a lower degradation rate and a smaller mass loss. The decomposition point at 300°C is also more pronounced. The acetone treatment had a minimal influence on the thermogravimetric behavior of both biomasses. Decomposition extends slightly further in the untreated sample, continuing to lose mass up to 600°C, although very slowly. The presence of extractives in the virgin wood and chestnut bark samples affects the decomposition behavior of the biomass. The TGA results suggest that detannized wood exhibits more rapid degradation at higher temperatures, which implies better combustibility and potential as a biofuel source. The improved thermal stability of the lignin-dominated DT and B samples indicates that these materials may be more suitable for high-temperature insulation materials or thermal barrier applications in biocomposite formulations. Additionally, the slow degradation of lignin in DT and B_noextr samples highlights the potential use of these materials in thermally stable biopolymer composites and high-performance carbon-based materials. The enhanced degradation of hemicellulose in detannized samples also supports their potential application in biochemical conversion processes, such as pyrolysis and gasification, where more controlled thermal breakdown is required for bio-oil or syngas production. The slow decomposition of untreated bark suggests its potential use in slow-burning biochar production, which could be utilized for soil amendments, carbon sequestration, or filtration applications. In contrast, the effect of extractives is less apparent in the detannized wood sample.

The calorific value represents the heat generated through the combustion of 1 kg of wood, and its determination is fundamental for evaluating both its energetic value and its flammability or potential to generate and propagate fires. This measure is dependent on various factors, including wood type, moisture content, age, orographic conditions and soil composition of its origin. The chemical properties of wood are believed to be influenced by these variables. The humidity of wood has a pronounced influence on its combustion efficiency (Vidrich, 1988). Wood extractives such as resins, waxes, oils, tannins and other phenolic substances have much higher heating values than cellulose and hemicellulose, and as previously stated, chestnut wood contains significant amounts of these compounds (Álvarez-Álvarez, 2018). The minimum difference in the calorific value of our samples could be attributed to the variation in the moisture content, as well as the carbon content. The fixed carbon content of biomass can be related to the calorific value since it has a positive effect on the energy potential of biomass (Özyuğuran and Yaman, 2017); however, in our samples, DT had the highest calorific value, while its average fixed carbon percentage was slightly lower than that in the VW. The high ash content in chestnut bark, due to its relatively high inorganic components (Poletto et al., 2012), naturally reduces its quality as a fuel (Álvarez-Álvarez, 2018). Therefore, the significantly lower ash content observed after the removal of extractives is crucial for future analysis and potential applications. The thermal properties of biomass can be strongly influenced by its chemical composition (Sebio-Punal et al., 2012). Another important consideration that influences the energetic value is the volatile materials that are released when organic substances are heated at high temperatures (Garcia et al, 2012). Volatile matter is the fraction of biomass that transforms into vapors during the pyrolysis process and condenses to produce bio-oil and non-condensable gaseous products (Ahmed et al, 2021). The correlation between the volatile matter in biomass and the heating value is complex because a higher percentage of volatile matter does not necessarily indicate a superior heating value, as some volatile matter components, such as CO2 and H2O, are noncombustible fractions (Özyuğuran and Yaman, 2017).

Table 3. Results of the various parameter measurements (moisture, ash content, volatile matter, fixed carbon and calorimetry)

Sample	**Average moisture content (%)**	**Calorific value (MJ/kg)**	**Average volatiles (%)**	**Average ash content (%)**	**Average fixed carbon (%)**	**Average dry volatiles (%)**	**Average dry ashes (%)**	**Average dry fixed carbon (%)**
Before the acetone removal of the extractives								
VW	8.32		71.83	0.59	19.27	78.34	0.64	21.01
DT	8.31		74.54	1.20	17.61	79.48	1.25	19.20
B	7.94		70.90	4.49	16.67	77.02	4.87	18.11
After the acetone removal of the extractives								
VW	9.61	16.63	68.95	0.23	21.22	76.28	0.26	23.47
DT	7.91	17.56	73.27	0.72	18.11	79.57	0.77	19.67
B	9.43	15.81	72.34	1.81	16.42	79.87	2.0	18.13
Extractives after acetone removal								
Extractives VW	8.45		59.84	0.17	31.54	65.36	0.19	34.45
Extractives DT	6.33		68.06	0.33	25.29	72.65	0.35	27.00
Extractives B	0.31		86.27	0.41	13.01	86.54	0.41	13.05

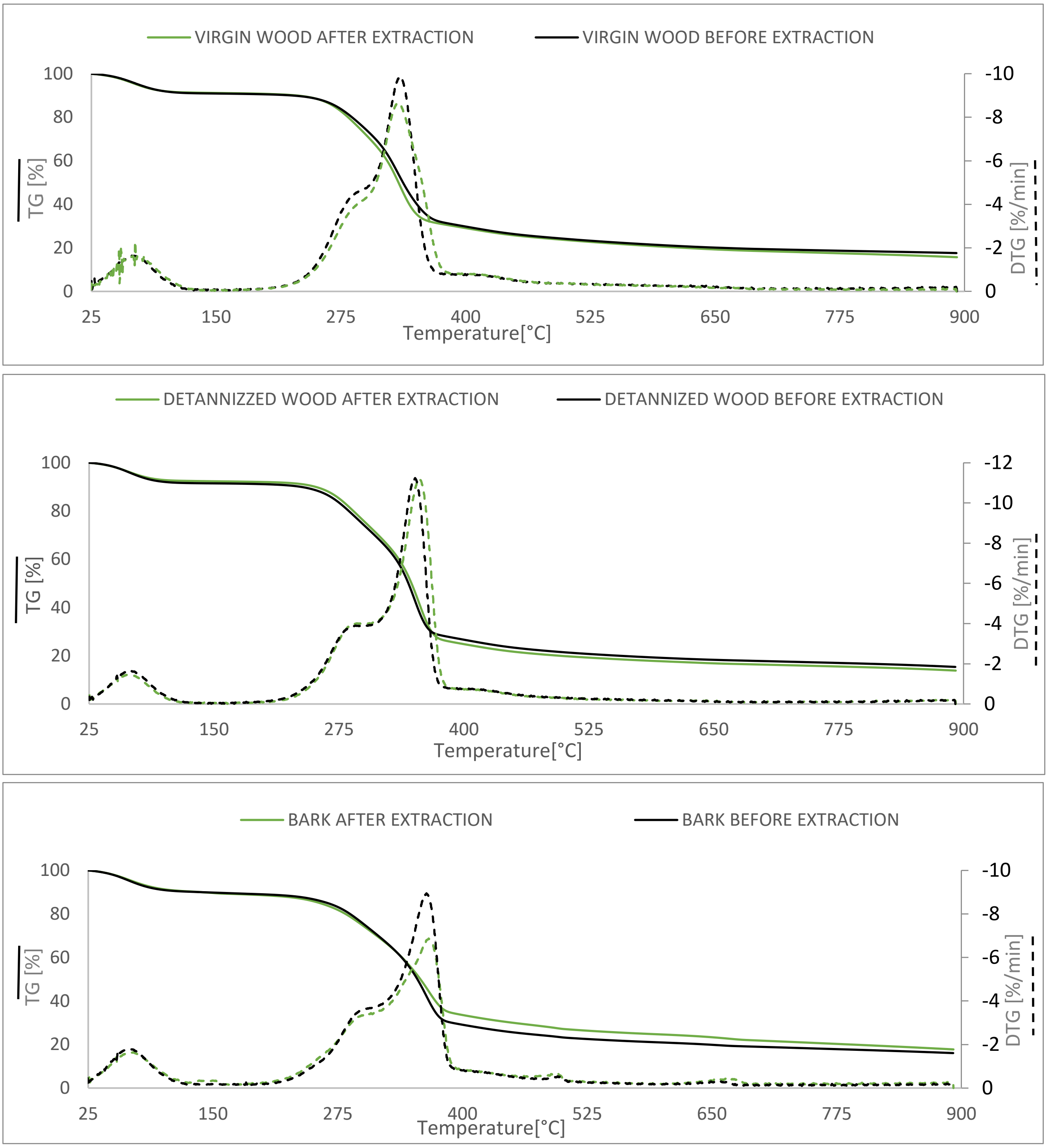

Figure 4. Thermogravimetric (TG) curves and derivative of thermogravimetric (DTG) curves of the three samples (VW, DT, B) before and after acetone extraction

4. CONCLUSION

Research and studies, such as that by Giovando et al. (2019), have extensively demonstrated the presence and potential utility of residual biomass compounds from the tannin industry. Understanding and efficiently utilizing these by-products are essential not only for maximizing the value of the extraction process but also for minimizing waste and promoting sustainability in industrial processes. This study delves into the significance of sweet chestnut in Europe, particularly within the context of Italy's forestry industry, and provides insights into the chemical and physical properties of chestnut wood residues before and after tannin extraction. A key focus is the possibility of utilizing chestnut wood residues, primarily in the tannin extraction industry, which exemplifies a circular economy within the forest-wood chain. Three different types of chestnut wood waste were analyzed: virgin wood waste (VW), chestnut wood bark (B), and detannized wood waste (DT). The variations in extractive content, molecular weights, and elemental composition highlight the potential for utilizing these residues in high-value applications, supporting sustainable development and circular economy concepts. The key findings of this study revealed several important aspects of chestnut wood and its bark. The virgin wood samples presented the highest percentage of extractives at 7.17%, whereas the bark had the lowest percentage at 2.5%. Despite undergoing an industrial extraction process, the detannized wood still retained a significant amount of extractives, measuring 4.02%. In terms of chemical composition, the lignin content remained relatively consistent across all the samples, with the bark showing the highest percentage. The cellulose and hemicellulose contents were also analyzed, revealing that, compared with virgin and detannized wood, bark had slightly lower concentrations. Additionally, the study assessed the thermal behavior and elemental composition, including the carbon and nitrogen contents, to evaluate the potential use of these residues as fuels or in other applications. In exhausted wood from tannin industry, lignin becomes more chemically accessible due to the removal of tannins, which likely alters the structural integrity of the biomass and enhances its potential for thermal processing and conversion into bio-based materials.

The insights gained can guide further research and development of sustainable utilization methods for these wood residues.

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