Feedstock pretreatment

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Table 1: Document information

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Table 2: Dissemination level of this report

Dissemination level of this report	
PU	Public

Table 3: List of Abbreviations

Abbreviation	Definition
EFG	Entrained flow gasification
TGA	Thermogravimetric analysis
DTG	Derivative thermogravimetry
FC	Fixed carbon

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EXECUTIVE SUMMARY

Deliverable D3.2 provides a comprehensive overview of the feedstock preparation and torrefaction processes for entrained flow gasification (EFG). It includes an analysis of feedstock composition, covering ultimate, proximate, and metals content. Small-scale torrefaction tests using thermobalance (TGA) equipment were conducted, yielding critical data on biochar carbon content, reaction kinetics, and volatile emissions. These tests helped to determine the optimal operating conditions—specifically temperature and residence time—that produce torrefied material with the desired properties. The results were further validated through experiments in a mini-lab pyrolysis plants, which produced torrefied biomass. Torrefaction was carried out in two types of reactors: a batch-load continuous mode reactor, and a continuous mode reactor without a batch (screw-type reactor). After feedstock preparation, all products were characterised using ultimate, proximate, and metals analysis. Additionally, detailed chemical analyses—including volatile content, ash content, calorific value, GC/MS analysis, and semi-online GC analysis for gases—were conducted and are presented in this deliverable. This process provides valuable insights into torrefaction conditions and product characteristics, helping to optimize feedstocks for future energy production applications.

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1 Torrefied biomass

Torrefied biomass refers to biomass that has undergone a thermal treatment process known as torrefaction, which enhances its physical and chemical properties for energy production. This process involves heating biomass at temperatures ranging from 200 °C to 350 °C in an inert or low-oxygen environment, resulting in a carbon-rich, brittle material with improved energy density, increased resistance to moisture, and enhanced combustion properties [1]. Torrefaction removes volatile compounds, decomposes hemicellulose, and partially alters cellulose, making the biomass more uniform in composition and easier to transport and store [2]. Torrefied biomass, with its improved fuel properties, is used as a renewable fuel in power plants, industrial boilers, and in pellet form for heating. It is also suitable for biofuel production via gasification and pyrolysis [3].

In the context of the GIFFT project, torrefied biomass will be employed as a pre-treatment method for low-value, sustainable biogenic residues and waste feedstocks. The inherent diversity of biomass presents challenges for efficient syngas production, but pre-treatment techniques like torrefaction enhance the physical and compositional uniformity of feedstocks, reducing tar formation and improving syngas yield [4]. Additionally, pre-processing biomass and waste increases the energy density per unit mass, reduces transport costs, and removes undesirable substances that could impede thermal conversion technologies.

Biomass used in an entrained flow gasifier must be milled into very fine particles to ensure efficient gasification. Raw biomass is often difficult to mill into such small sizes due to its fibrous and tough nature. However, torrefied biomass, with its more brittle and uniform composition, is much easier to grind into the required fine particles [5]. This improved grindability enhances the efficiency of the gasification process, making torrefied biomass an ideal candidate for use in an entrained flow gasifier. Although biomass pre-treatment is a key component of the GIFFT concept, it is not the primary focus of development within the project. However, it will be crucial for ensuring the overall feasibility of the concept, with torrefied biomass specifically tested in an entrained flow gasifier for syngas production.

2 Feedstock characterisation

Biomass feedstock competition demands improved knowledge about their availability and characteristics, especially for low-cost biogenic residues and waste materials. Biomass-based fuel commercial suitability depends greatly on obtaining inexpensive feedstocks while maintaining an effective supply chain framework. The most economical method utilizes nearby feedstock sources that reduce logistical needs, possess processing compatibility, and simplified pre-treatment capabilities through mechanical and thermal processes. Among recognizable feasible local feedstocks are logging residues that function as available biomass sources in Northern Europe, specific German regions, and the Baltic countries, along with hay as agricultural waste and waste lignin from industrial production. An assessment of wood combined with straw and lignin wastes through thermal decomposition, ultimate and proximate analyses, and metals assessment including sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), lead (Pb), chlorine (Cl), potassium (K), calcium (Ca), titanium (Ti), chromium (Cr), manganese (Mn), iron (Fe), nickel (Ni), copper (Cu) and zinc (Zn) evaluation is needed.

Before each experiment, the drying plates receive moisture content measurements where their weights are recorded. The distribution of samples across the plates proceeds after establishing the plate mass, whereby the entire plate surface should receive at least a thin and uniform layer. The drying oven maintains a temperature of 105 ± 2 °C for a duration until successive mass measurements show no changes for a period of 60 minutes. Scientists determine stability when successive measurement variations stay under 0.2% of the recorded value. The moisture content percentage of the sample can be determined by dividing the mass loss from its initial mass value.

To determine the ash content of the material, ash plates are used, which are weighed without any sample at the start of the experiment and the scale reading is recorded. The mass of the sample used for the tests shall be at least 1 g, which shall be placed in the ash trays and weighed. To determine the ash content, the material must be fully oxidised, so air is used as the initial oxidant. Oxidation is carried out for 60 minutes at an initial temperature of 250 ℃ and for at least 120 minutes in an oven heated to 550 ℃. The experiment is considered complete when the mass of the sample does not change more than 0.5 mg in the last 30 minutes. The ash content of the sample was calculated from the mass loss from the added sample by measuring the residual mass of the sample and expressing it as a percentage of mass. Ash analysis was carried out in accordance with standard LST EN 15403: 2011. The percentage of fixed carbon (FC) formed is calculated by considering ash, volatile compounds and moisture.

A thermal analysis method determines the volatile compound quantities present in raw materials assessed for this study. A ceramic crucible is used to contain a known mass of thoroughly pulverized dried samples that shows a uniform distribution of particles under 1 mm. After weighing the crucible, which contains the sample, it is placed into an oven set at 900 ± 10 °C within ambient atmospheric pressure. After the oven is heated, the experimenter removes the crucible containing the sample while it slowly reaches room temperature. The experimental period lasts for 7 minutes with an accepted time window of 5 seconds. A volatile compound evaluation takes place through a precise assessment of lost mass. The tests followed all requirements presented in LST EN 15148:2010.

A Dionex ISC 5000 ion-exchange chromatograph measured chlorine concentration within tested samples. The analysis required drying and later crushing and grinding the samples into 1 mm particles before subjecting them to a 35-bar oxygen-filled calorimetric bomb. The flask received passed gas oxygen through deionized water, which allowed dissolved chlorine from the gas to form water solutions. A Dionex ISC 5000 ion-exchange chromatograph evaluated the solution to determine chlorine levels by applying a pre-made calibration curve. Throughout their performance, the experiments followed the testing requirements stated in LST EN 15408:2011 and LST EN 15289:2010.

The Flash 2000 analyser measures quantitative elements in materials through C, H, N, and S determinations. A preparatory step before analysis includes drying the samples while reducing them to 1 mm particles and thoroughly mixing them. Total oxidation of all samples happens when they are treated in oxygen-rich conditions. Oxygen can be calculated by subtracting the measured element values together with ash content from total concentrations. The methods for determining carbon, hydrogen and nitrogen elements follow the requirements established in LST EN 15407:2011 and LST EN 15104:2011.

The characterization of sample thermals properties together with their high-temperature characteristics made use of the Netzsch STA 449 F3 Jupiter thermal analysis system to perform thermogravimetric analysis (TGA). A weight measurement of a blank Al_2O_3 crucible began this analysis. The recorded weight measure represented its initial mass. The sample weighing 9.5 ± 0.2 mg was transferred to the crucible before the system received this substance for analysis. The thermal analysis program was chosen properly then the temperature increased to 900 °C using a heating rate of 20 °C/min. The experimental period relied on N_2 as a carrier gas that operated at 60 mL/min. Through the solenoid valve the balance system received the gas which was directed to the thermal chamber for experimental execution. Because infrared heat loss presents a challenge in high-temp applications the experiment used radiation shields installed on the balance system's sample holder.

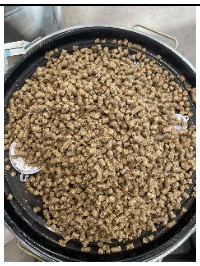
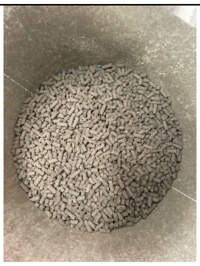
2.1 Ultimate, proximate, and metal's analysis

The material suitability assessment for syngas production through entrained flow gasification requires performing a composition evaluation. The raw material composition analysis is presented in Table 2.1.

The elemental composition and proximate analysis of wood, straw, and lignin reveal significant differences in their thermal properties and potential applications in bioenergy and material processing [6]. The standards of moisture among feedstocks differ with straw (4.42 wt%) maintaining the highest percentage and wood (2.09 wt%) showing the lowest values, while lignin reaches 3.98 wt%. The thermal decomposition rate of straw and lignin can be affected by their higher moisture levels since it increases the amount of energy needed to dry materials before combustion or pyrolysis [7].

The values of volatile matter basically determine how easily biomass materials will combust. Among the three materials wood possesses 77.63 wt% in volatile content while straw contains 68.48 wt% and lignin exhibits the lowest amount at 46.22 wt%. Reductive thermal breakdown speeds up in materials containing significant volatile contents such as wood and straw hence these substances work best for gasification and pyrolysis operations [8]. The thermal stability of lignin is high due to strong polymeric matrix into its structure, leading to the slower decomposition processes.

Table 2.1. *Wood, straw and lignin proximate analysis.*

Parameter	Wood	Straw	Lignin
Picture of the feedstock			
Moisture, wt%	2.09	4.42	3.98
Volatiles, wt%	77.63	68.48	46.22
Fixed carbon, wt%	18.04	16.78	28.71
Ash, wt%	2.24	10.32	21.09

The investigation of fixed carbon and ash indicates that lignin produces high amounts of solids residual while requiring an extended burning duration: it features 28.71 wt% fixed carbon. Wood and straw demonstrate lower fixed carbon content at 18.04 wt% and 16.78 wt%, respectively. Due to its extensive fixed carbon content, in this case, lignin is an effective raw material for carbon-based product development and biochar manufacturing.

Moreover, lignin contains 21.09 wt% ash content, which exceeds straw's 10.32 wt% content, whereas wood features only 2.24 wt%. The high ash levels in materials can cause system operational issues during it application in various energy-recovery processes [9]. Due to its minimal ash content, wood is an optimal fuel source for thermal operations relative to both straw and lignin, as these feedstocks demand supplementary ash processing or handling methods [10].

The proximate analysis of the main elemental components is presented in Table 2.2. The data provided compares the elemental composition and energy content of the selected biomass samples. As can be seen from the results, each material has distinct characteristics that influence its potential applications, environmental impact, and energy production [11]. Lignin contains the highest percentage of elemental carbon at 49.41%, followed by wood at 44.69%, and straw at 42.71%. Moreover, wood and straw samples have almost similar hydrogen percentages, with 5.97% and 5.82%, respectively, while lignin contains less hydrogen at 4.20%, which affects its energy density and combustion characteristics. Straw has the highest nitrogen content at 0.89%, slightly higher than wood (0.18%) and lignin (0.72%), and the higher nitrogen content can lead to increased NO_x emissions during the thermal treatment. The Sulphur (S) and Chlorine (Cl) were detected in small quantities across all materials. Straw has a slightly higher sulphur content at 0.09%, while lignin has a higher sulphur content than wood and straw. Chlorine levels are minimal in all samples. Calculated by difference (based on eq. 1), oxygen content is highest in wood at 46.89%, followed by straw at 40.12%, and lowest in lignin at 24.23%.

$$O_2 = 100\% - C - H - N - S - Cl - Ash \qquad (1)$$

Moreover, the lower and higher heating values (LHV and HHV) are also presented. It was investigated that lignin has the highest energy content, with an LHV of 17,996 kJ/kg and an HHV of 18,906 kJ/kg. Wood follows closely with an LHV of 16,895 kJ/kg and an HHV of 18,201 kJ/kg. Straw has the lowest energy content, with an LHV of 15,070 kJ/kg and an HHV of 16,341 kJ/kg.

In summary, lignin stands out for its high carbon content and energy density, making it a promising material for energy applications. However, its lower oxygen content and higher sulphur levels require careful consideration in gasification processes to minimize environmental impacts. Wood and straw, while less energy-dense, offer alternative biomass options with different environmental profiles.

Table 2.2. *Wood, straw and lignin proximate analysis and heating values.*

Parameter	Wood	Straw	Lignin
C, %	44.69	42.71	49.41
H, %	5.97	5.82	4.2
N, %	0.18	0.89	0.72
S, %	0.02	0.09	0.34
Cl, %	0.01	0.05	0.01
O (diff), %	46.89	40.12	24.23
LHV, kj/kg	16895	15070	17996
HHV, kj/kg	18201	16341	18906

The evaluation of concentrations of metals is presented in Table 2.3, and visual presentation is depicted in Figure 1. As could be seen from the results, wood and straw has a lower concentration in most of the elements, except potassium. In all of the samples, no sulphur, fluorine and bromide were detected, meaning these elements are absent, or below detection limit. The highest concentrations of the elements in the wood sample are calcium, reaching 1008.06 mg/kg, iron, reaching 483.24 mg/kg, potassium, reaching 722.91 mg/kg, and chlorine, reaching 280 mg/kg.

Table 2.3. *Metal analysis of the raw material*

Element	Wood	Straw	Lignin
Al, mg/kg	20.29	875.43	4865.54
Ca, mg/kg	1008.69	2168.08	7776.46
Cr, mg/kg	0.47	4.21	90.05
Cu, mg/kg	0.95	2.96	225.38
Fe, mg/kg	483.24	879.40	5334.21
K, mg/kg	722.91	8272.61	2788.43
Mg, mg/kg	161.65	917.19	1630.76
Mn, mg/kg	29.69	44.02	266.00
Na, mg/kg	47.14	214.39	1520.72
Ni, mg/kg	0.00	0.00	14.99
Pb, mg/kg	4.37	1.45	9.80
Si, mg/kg	71.23	10109.89	26177.61
Ti, mg/kg	26.08	55.60	313.29
Zn, mg/kg	9.29	11.89	183.67
Cl, mg/kg	280.00	440.00	90.00
S, mg/kg	0.00	0.00	0.00
F, mg/kg	0.00	0.00	0.00
Br, mg/kg	0.00	0.00	0.00

The highest concentrations of elements in the straw sample are aluminium, reaching 875.43 mg/kg, calcium, reaching 2168.08 mg/kg, iron, which reaches 879.4 mg/kg, potassium - 8272.61 mg/kg, magnesium - 917.19 mg/kg, silicon - 10109.89, and chlorine - 440 mg/kg. The most significant concentrations were detected in the lignin samples, with an element of aluminium, calcium, iron, potassium, magnesium, sodium, and silicon, with a value of 4865.54 mg/kg, 7776.46 mg/kg, 5334.21, mg/kg, 2788.43 mg/kg, 1520.72 mg/kg, and 26177.61 mg/kg, respectively. The predominant retention of metals in lignin takes place inside the char during the gasification or combustion phase [12], because it impacts the properties of whole process, forcing the formation of slags in the reactors.

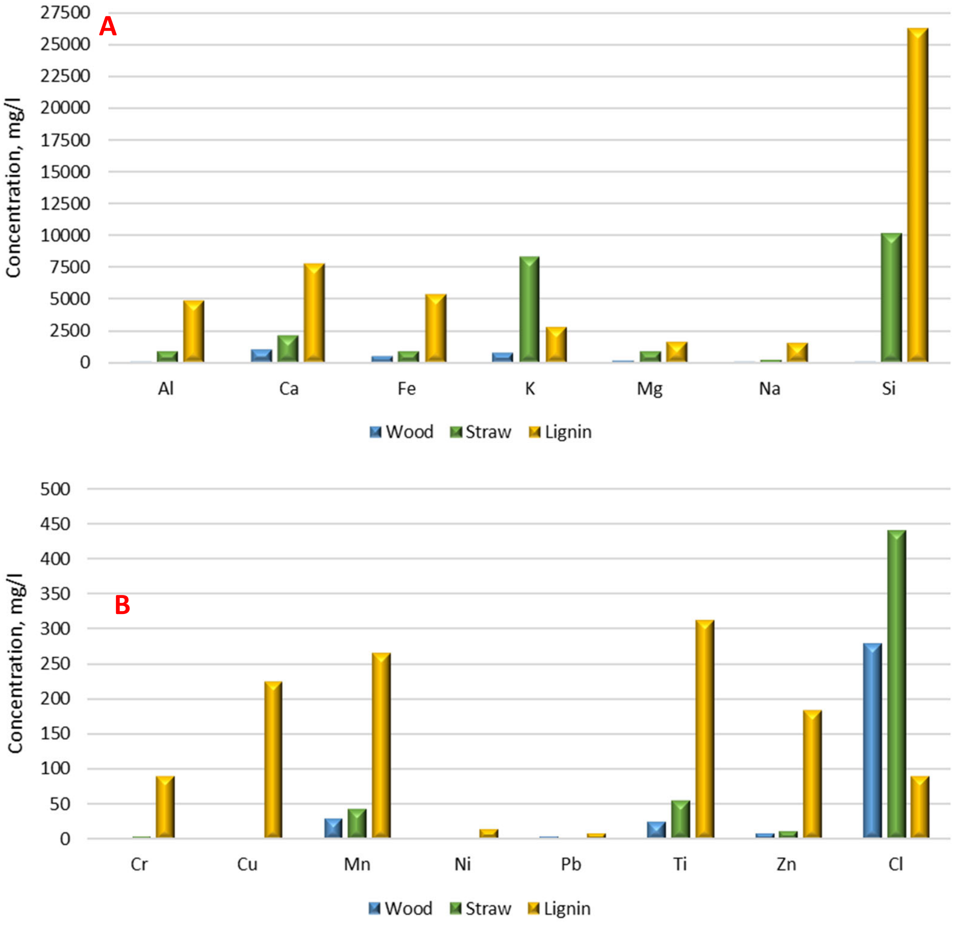


Figure 1. Elements concentrations in wood, straw, and lignin samples. A - concentrations above 500 mg/kg; B - concentrations below 500 mg/kg.

2.2 Thermal analysis at a micro scale

Thermogravimetric analysis (TGA) is a crucial analytical technique for evaluating biomass feedstocks' thermal decomposition and stability intended for torrefaction. TGA provides critical insights into biomass's thermal behaviour and mass loss dynamics, which are essential for optimizing torrefaction conditions and ensuring efficient processing. Figure 2 represents the TGA (A) and DTG (B) profiles of three selected samples.

The TGA curves (Figure A) illustrate biomass's mass loss as a temperature function. The weight of all samples up to 150 °C temperature is lower slightly because of moisture removal. The mass loss of straw exceeds the other samples in this temperature range, indicating straw holds greater water content than lignin and wood. The thermal decomposition of hemicellulose and cellulose causes major weight reduction within the temperature range of 200–400 °C. Wood samples and straws degrade rapidly by temperature, but straw decomposes earlier, suggesting that straw has less thermal stability.

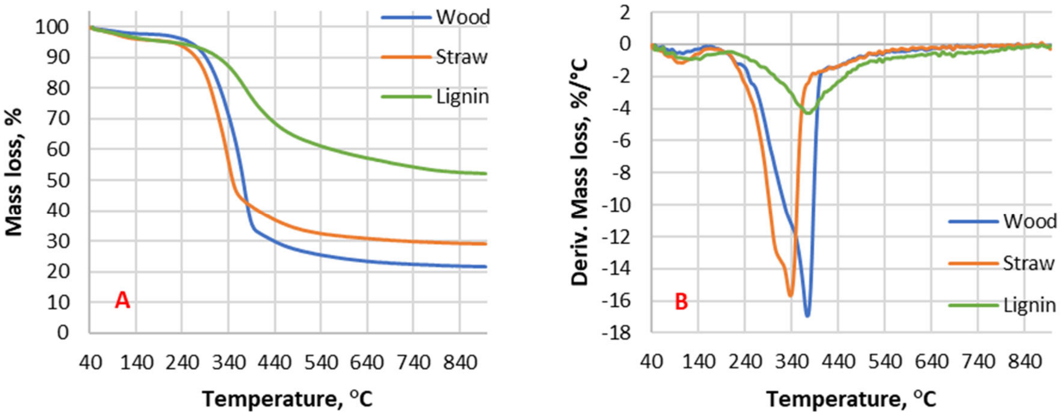


Figure 2. TGA (A) – DTG (B) curves of wood, straw and lignin

The decomposition process of lignin unfolds throughout a wide temperature span because the substance demonstrates strong thermal resistance and an elaborate decomposition pathway. Most of the remaining material contains fixed carbon and ash when the temperature exceeds 400 °C. Because lignin features a high fixed carbon amount and small volatile matter content, it maintains the most significant remaining substance fraction. The overall mass loss for straw exceeds all other samples, which matches its volatile matter percentage combined with high ash content. Figure B shows the mass loss rate against temperature relationships to display decomposition reaction speeds. According to thermal decomposition analysis, the fast cellulose destruction occurs between 300–350 °C in wood and straw materials. The thermal stability of straw proves to be lower than wood since it shows a slightly reduced peak temperature. The degradation process of lignin extends across broad temperature ranges, which causes its degradation rate to appear at a broad but shallow peak. Because lignin contains a significant amount of stable carbon, it demonstrates slower decomposition than other biomass components in heating environments.

3 Torrefaction procedure

The torrefaction procedure must be applied for the biomass to apply the desired feedstock parameters, such as decreased the number of volatiles, increasing the energy density and tough, easily crushable, and water-repellent structure [13]. For that reason, firstly, it was used continuous-mode batch-load reactor for straw and lignin biomass, but this system has a lot of disadvantages, which will be wider presented in next sections. For that reason, the second version of torrefaction device was constructed and applied for wood biomass. The wider characteristics and descriptions are provided in the sections 3.1 and 3.2.

3.1 Experimental setup for straw and lignin torrefaction

To prepare torrefied biomass, the thermal treatment keeping an inert atmosphere must be applied. In the context of this project, the continuous-mode batch-load reactor functions as a torrefaction bench, and visual scheme is depicted in Figure 3. This reactor consists of feedstock bunker, screw type-cooled feeding device, main high-temperature reactor and solid products removal systems. Moreover, on the top the reactor there are gaseous products removal valve, directing the flow to combustion chamber. The feeding device supplies the feedstock from the bunker into the reactor for about 10 kg per batch. The reactor was preheated up to the temperature of 240 °C for straw, and 330 °C for lignin. These temperatures were selected based on the product quality and characteristics: if the temperature is too high, the pyrolysis process starts, and the majority of the volatiles evaporate from the feedstock; vice versa, not enough volatiles are emitted, meaning the biomass structure is not tough enough with increased grindability. Moreover, the retention time of the feedstock in the high-temperature reactor plays a crucial role. Keeping the feedstock too long in the reactor leads to the fixed carbon formation. Nevertheless, if the retention time is too short, just a small part of the volatiles emits, not reaching the torrefied biomass specifications, which was mentioned above. For that reason, the optimum retention time was determined to be 4 hours for straw, and 2 hours for lignin. Nevertheless, the solid product removal system efficiently recovers torrefied biomass from the process, so the operation can be performed in continue mode without any interruption. An inert gas supply system using nitrogen is connected to the ash removal device bunker, what brings continuous flow to establish oxygen-free process conditions required for thermal degradation control without oxidation. A control system and instrumentation maintenance several critical process variables including temperature along with gas stream rates and residence duration for sustaining reliable operation, while the reactor produces torrefied biomass continuously.

The main operational difficulty of this reactor involves controlling the thermal energy output. The ongoing torrefaction reaction produces heat inside the system which results in temperature elevation because its process is exothermic by nature. The temperature increase affects torrefied biomass properties in negative ways. Operation difficulties mainly stemmed from controlling the reaction process particularly when processing large biomass amounts. The process control system faced challenges which resulted in unstable conditions among different runs resulting in variable product outcomes. A different torrefaction reactor was developed specifically to overcome stability issues during the manufacturing process under laboratory testing conditions.

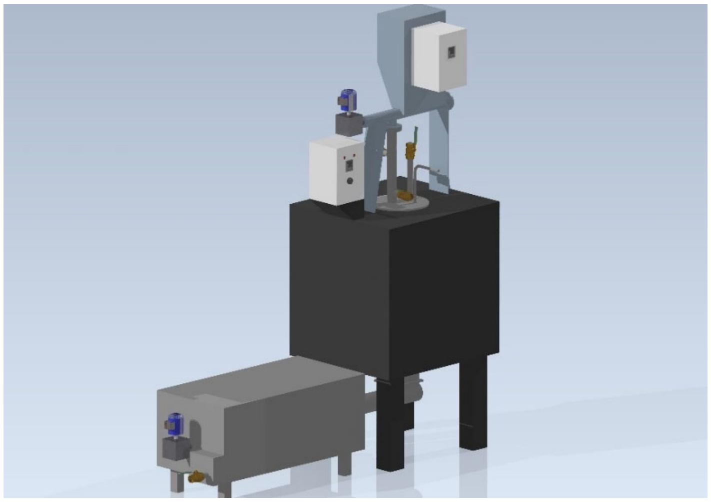


Figure 3. Load batch continuous mode torrefaction reactor

3.2 Experimental setup for wood torrefaction

The scheme of the continuous mode torrefaction reactor is presented in Figure 4. This type of reactor consists of these main segments: bunker, screw-type feeding device, screw-type reactor, and product collection bunker. As can be seen from the visualisation, the feedstock is placed into the bunker on the top of the reactor. Then, by screw-type feeder, biomass is supplied to the high temperature reactor chamber, with a feeding rate at 2.3 kg/h. This reactor has two chambers, one above another, connected in series, with 4 independently controlled heating zones, as presented in the picture. Based on the product characterisation for the wood torrefaction procedure, it was determined to have an optimum regime: 1-2 zones – 210 °C, and 3-4 zones – 200 °C.

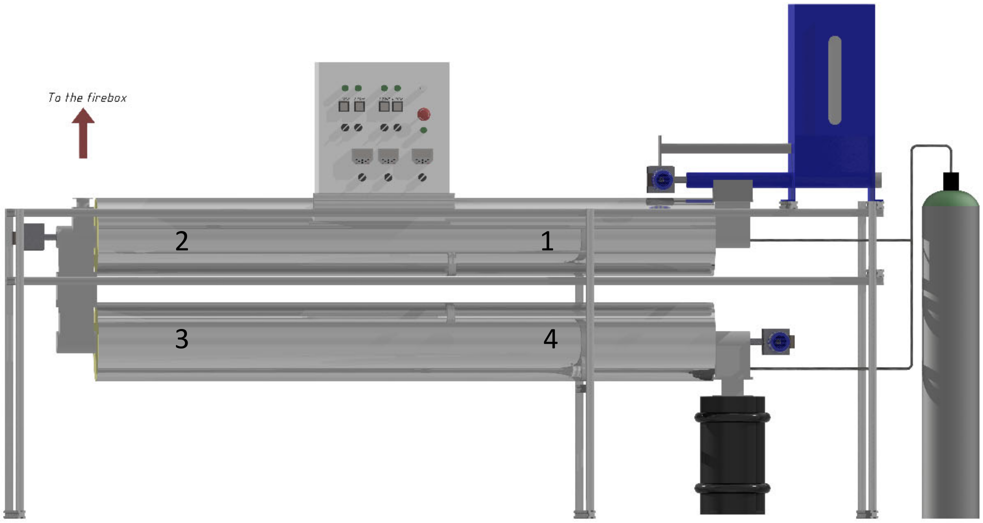


Figure 4. Screw-type continuous mode torrefaction reactor

Both chambers are connected to nitrogen gasses to create an inert atmosphere with a flow rate of 8 l/min. Moreover, on the reactor's left side, there is one exhaust valve for the removal of emitted gasses, which are supplied to the combustion chamber. Feedstock passes both reactors and enters the solid products collection bunker. The quality of the product is also significantly influenced by the retention time, which was 12.5 min., and the temperature inside the reactor.

This type of reactor has a considerable advantage over the load-batch reactor because the feedstock always moves inside the reactor, avoiding overheating in contact with another biomass particle. By changing the retention time (by the speed of the rotational screw), and the temperatures in different zones, the optimum parameters were achieved, which will be presented in the next section.

4 Torrefied biomass analysis

4.1 Proximate analysis

To evaluate the quality of pretreated biomass, it is very important to investigate its ultimate and proximate characteristics in terms of torrefaction regimes' influence on the whole process. The proximate analysis results are presented in Table 4.1.

The analysis of the torrefied biomass shows important composition differences as wood samples demonstrate distinct features from straw and lignin waste materials. It is related with the raw, untreated biomass composition. During the torrefaction procedure there was achieved two main objectives: increased energy density and stability through volatile matter reduction, while raising fixed carbon percentages in biomass materials. Torrefaction of wood results in maximum volatile preservation at 52.53%, while straw reaches 49.24% and lignin maintains 36.89% volatiles. The lower volatile matter loss rate of torrefied wood compared to torrefied straw and lignin produces advantageous results regarding energy content maintenance. The fixed carbon content of torrefied wood reaches 43.01%, thereby making it suitable as a fuel source because it provides more energy density than other selected biomasses. Lignin and straw contain lower fixed carbon levels, reaching 38.97% with lignin, while straw maintains 37.84%. The samples display different levels of ash in proportion to each other. Torrefied lignin's high ash content at 24.14% represents a danger during the EFG processes, because slagging and fouling reactions become probable. The ash content in straw reaches 12.92%, while wood contains a lower percentage of 4.46%.

Table 4.1. *Proximate analysis of torrefied wood, straw and lignin*

Parameter	**Torrefied wood**	**Torrefied straw**	**Torrefied lignin**
Picture of the feedstock			
Moisture, wt%	-	-	-
Volatiles, wt%	52.53	49.24	36.89
Fixed carbon, wt%	43.01	37.84	38.97
Ash, wt%	4.46	12.92	24.14

In comparison with raw biomass, the number of volatiles were reduced by around 27.19 wt% with wood, 23.66 wt% with straw, and 13.31 wt% with lignin. It could be explained by the feedstock composition: wood has the highest amount of cellulose and hemicellulose in its structure, leading to the lower energy demand to decompose these compounds, in the form of volatile fraction. While the amount of these molecules decreases, the demand of energy increases, leading to the lower volatile's removal by the torrefaction procedure.

The fixed carbon content increases by 24.97 wt% in wood biomass, 21.06 wt% in straw, and 10.26 wt% in lignin during torrefaction. The composition of cellulose along with hemicellulose and lignin in wood makes volatile substances release possible, while maintaining a high proportion of carbon in the remaining solid structure leading to its highest amount of fixed carbon, with a value of 24.97 wt%. The amount of fixed carbon in straw remained at 21.06 wt% possibly because of its high initial ash content together with low lignin content which prevents carbon retention. The mass loss of 10.26 wt% from lignin remains low because this organic compound exists as a thermostable carbon-rich group that resists temperature change. The higher fixed carbon levels within torrefied biomass boost its calorific values along with its thermochemical efficiency levels thus creating an energy-dense fuel material. The transformation plays a vital role in upgrading biomass quality because it benefits bioenergy applications as well as thermochemical conversion operations.

4.2 Ultimate analysis

An analysis of elemental composition and energy content exists in the data regarding torrefied wood along with straw and lignin wastes. The ultimate analysis results of the torrefied biomass are presented in Table 4.2. An analysis of elemental composition and energy content exists in the data regarding torrefied wood along with straw and lignin wastes. Of the analysed materials torrefied wood demonstrates the highest carbon content reaching 68.50% followed by straw and lignin which show about 54% carbon content. Typical for torrefaction processes is this rise in carbon content because heat decomposes hydrogen and oxygen-containing compounds to create carbon-rich end products [5], [14]. Torrefied wood contains 4.11% hydrogen, while straw – 4.99%, and lignin – 4.18%. The amount of nitrogen differs among the samples where straw has 1.12%, but wood has only 0.34% and lignin contains 0.80%. The highest sulphur content was determined in lignin, where the value reaches 0.49% compared to wood and straw levels, which is negligible. Straw contains the highest amount (29.90%) of oxygen while lignin possesses the lowest quantity (14.86%) based on difference calculations.

Torrefied biomass exhibits its stored energy through assessment of lower heating value (LHV) and higher heating value (HHV). The energy density of torrefied wood surpasses straw and lignin because its LHV reaches 23,118 kJ/kg and its HHV reaches 24,011 kJ/kg. The enhanced carbon content together with reduced oxygen content found in torrefied wood matches well with its energy application needs [15][14]. Both straw and lignin show lower heating values than torrefied wood where straw reaches 21,380 kJ/kg, and lignin – 19,088 kJ/kg. Torrefaction treatment improves biomass's physical capabilities including its grindability and hydrophobicity which enhances its stability while improving handling capabilities [14][15]. Torrefying reduces moisture while raising carbon content thus creating combustible materials that resemble coal in power sector and gasification operations [5] [15].

Table 4.2. *Ultimate analysis of torrefied wood, straw and lignin*

Parameter	Torrefied wood	Torrefied straw	Torrefied lignin
C, %	68.5	54.18	54.25
H, %	4.11	4.99	4.18
N, %	0.34	1.12	0.8
S, %	0.05	0.08	0.49
Cl, %	n.d.*	0.03	0.02
O (diff), %	25.3	29.9	14.86
LHV, kj/kg	23118	21380	19088
HHV, kj/kg	24011	22454	19988

*n.d. – not detected

The wider analysis of the elements in the torrefied wood, straw and lignin samples are presented in Table 4.3, and visualisation is depicted in Figure 5. One of the key consequences of torrefaction is the changes of elemental composition through its concentrations due to the removal of moisture and volatile compounds. Part of the organic volatiles are removed, leading to the concentrations increasement of the elements in the torrefied samples.

The inorganic elements in torrefied wood remain at moderate levels while compared to both straw and lignin. The major elements of torrefied wood include calcium at 2072.41 mg/kg, iron at 1390.99 mg/kg, potassium at 1397.41 mg/kg, and magnesium at 343.11 mg/kg. Torrefied wood possesses less ash-forming potential than straw and lignin yet present considerable increases in metal content especially copper (19.44 mg/kg), zinc (35.20 mg/kg), and chromium (8.52 mg/kg) following torrefaction due to concentration effects. The low 60.00 mg/kg chlorine level in torrefied wood presents attractive characteristics for combustion or gasification systems because it reduces the risks of corrosion and slagging damage.

The potassium content of torrefied straw exceeds other substances by reaching 18494.90 mg/kg which marks the highest concentration detected among all tested samples. The high quantities of silicon (10650.16 mg/kg) and calcium (3080.38 mg/kg) together with potassium indicate that ash-related combustion problems of slagging and fouling could occur. The presence of chlorine at 310.00 mg/kg creates equipment corrosion risks and emission concerns even though copper at 6.25 mg/kg and zinc at 10.33 mg/kg exist at moderate levels in torrefied straw. Straw provides numerous benefits because it remains an abundant biomass source with high energy content yet requires specific management during pre-treatment before using it in thermal processes.

The concentrations of all measured elements are the highest in torrefied lignin. The ash-forming potential of torrefied lignin is very high because silicon (27204.30 mg/kg) surpasses calcium (9502.03 mg/kg) and iron (7582.30 mg/kg) in its elemental composition. Significant traces of metals are found in torrefied lignin due to copper levels at 331.99 mg/kg combined with zinc at 231.69 mg/kg and aluminum at 5852.12 mg/kg and chromium at 93.11 mg/kg. Torrefied lignin contains traces of industrial processing residues based on its measured values. The chlorine content at 190.00 mg/kg in the material has slightly higher levels than wood but lower than straw which leads to moderate risks when using it in thermal treatment. High-energy lignin used as fuel requires controlled management because it contains metals that affect environmental safety.

Made comparison between raw and torrefied biomass revealed, that the thermal treatment increased the concentrations of the elements, meaning, that part of the volatiles was removed, increasing the residuals (metals) concentrations. Torrefaction of the three biomass varieties leads to major element enrichment across the board particularly for metals Al, Ca, Fe, Mg and K. The torrefaction process leads to iron element concentration growth from 483.24 mg/kg to 1390.99 mg/kg in wood and calcium element concentration doubles from 1008.69 mg/kg to 2072.41 mg/kg. The element concentration of potassium shows strong increase in straw samples (8272.61 mg/kg to 18494.90 mg/kg) while iron element concentration shows much stronger increase in lignin samples (5334.21 mg/kg to 7582.30 mg/kg).

Table 4.3. *The element's analysis of the torrefied wood, straw, and lignin samples.*

	Torrefied wood	Torrefied straw	Torrefied lignin
Al, mg/kg	53.06	1064.99	5852.12
Ca, mg/kg	2072.41	3080.38	9502.03
Cr, mg/kg	8.52	7.14	93.11
Cu, mg/kg	19.44	6.25	331.99
Fe, mg/kg	1390.99	1282.19	7582.30
K, mg/kg	1397.41	18494.90	4074.44
Mg, mg/kg	343.11	1496.32	1845.37
Mn, mg/kg	55.98	63.75	322.40
Na, mg/kg	159.70	298.86	1539.37
Ni, mg/kg	12.26	4.24	15.88
Pb, mg/kg	10.16	5.97	12.76
Si, mg/kg	114.12	10650.16	27204.30
Ti, mg/kg	61.35	61.07	446.81
Zn, mg/kg	35.20	10.33	231.69
Cl, mg/kg	60.00	310.00	190.00
S, mg/kg	0.00	0.00	0.00
F, mg/kg	0.00	0.00	0.00
Br, mg/kg	0.00	0.00	0.00

The torrefaction procedure decrease the concentration of chlorine content, mainly for wood and straw materials, from 280 mg/kg to 60 mg/kg and 440 mg/kg to 310 mg/kg respectively. The reduction of chlorine content becomes important in thermochemical conversion applications because this element contributes to system corrosion in combustion or gasification environments. The Cl content of lignin changes slightly during torrefaction when processing: from 90 mg/kg to 190 mg/kg, but the feedstock properties and process factors could affect the final results. Torrefaction treatment elevates ash-forming elements Si, Al and Fe concentrations while this produces unfavorable conditions for combustion or gasification performance due to increased accumulation of ash which leads to slag formation.

The torrefaction process enhances biomass energy density and hydrophobic properties so it becomes compatible further gasification applications, in the context of this project. However, the concurrent increase in inorganic and potentially problematic elements such as heavy metals and ash-forming compounds necessitates a comprehensive understanding of feedstock behavior post-torrefaction. These results show that better fuel characteristics result from torrefaction but produce higher inorganic material loads which have to be carefully managed during biomass conversion system design and operation.

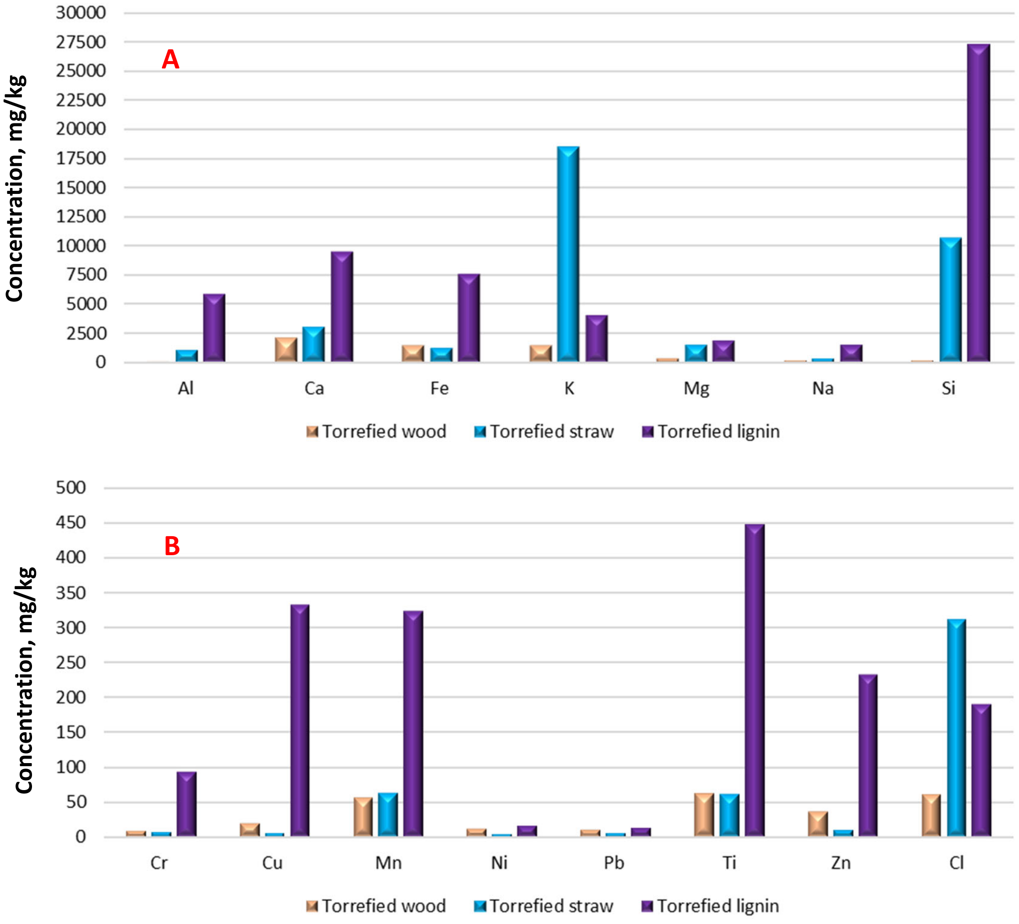


Figure 5. Elements distribution in torrefied wood, straw and lignin samples. A - concentrations above 500 mg/kg; B - concentrations below 500 mg/kg.

4.3 Torrefaction parameters influence for the product quality.

Feedstock’s retention time and the temperature are main parameters, which determines the quality of the torrefied biomass. Temperature defines which chemical bonds break, and what number of volatile compounds are released. Basically, temperature controls the ratio between the volatiles and residuals in the product material. Retention time also plays a crucial role in determining the degree of thermal degradation and decomposition. The longer biomass stays in the hot zone, the more extensively decomposition is. Moreover, short retention time partially removes the moisture and light volatiles, while the longer presence leads to the deeper breakdown of biomass components (cellulose, hemicellulose and lignin). So, the influence of these two parameters is very important, trying to achieve preferable products composition. The retention time influence for the number of volatiles at the constant, 200 °C temperature, is presented in the Figure 6.

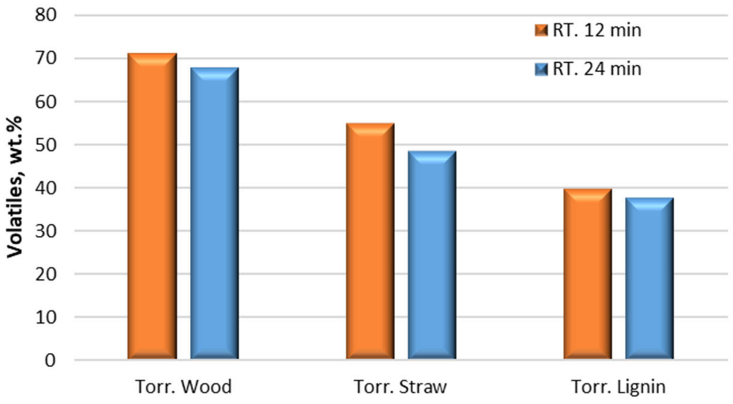


Figure 6. Retention time influence for the amount of volatiles

The bar chart demonstrates how three types of torrefied biomass consisting of wood, straw, and lignin respond to changes in their retention time, impacting their volatile matter content. A comparison was made between retention times that amounted to 12 minutes (orange bars) and 24 minutes (blue bars). The volatile content measurement decreased when the retention time was extended in all testing scenarios. In case of torryfied wood, the amount of volatiles content decreased from 71% to 66%, straw decreased from 54% to 48%, and lignin decreased from 39% to 37%. As could be seen from the results, the impact of the retention time for the torrefied biomass number of volatiles are minor, meaning, that the biggest impact are made by the selection of torrefaction temperature. It may be explained by the fact, that prolonged retention allows for more complete reactions, but does not significantly alter the fundamental chemical transformations, initiated by temperature. J. I. Orisaleye et al. [16] investigated that the increased retention time from 30 to 90 min. with agricultural residues has only marginal effect on the quality of the products and calorific value, compared with an increased temperature from 200 to 280 °C [17]. At lower temperatures (e.g., ≤230 °C), even extended retention times fail to achieve the same level of thermal decomposition observed at higher temperatures [18]. For that reason, the temperature influence for the number of volatiles at the constant, 12 min retentione time, is presented in the Figure 7.

As could be seen from the results, the experiments were performed over three temperatures: 200 °C (blue bar), 250 °C (orange bar), and 300 °C (grey bar). The analysis reveals, that an increase in torrefaction temperature leads to a substantial reduction of volatile content with all three types of biomass. The volatile content of torrefied wood at 200 °C reaches 70%, while the increased temperature decreased it to 45% at 250 °C, and at 300 °C it reaches 24%. The volatile fraction in straw decreased from 55% at the beginning at 200 °C to 30% at 250 °C, and ended at approximately 22% at 300 °C. The percentage of lignin reduces while performing to a minor degree from 40% (200 °C) down to 28% (250 °C), then ending at 21% (300 °C). The solid transformation into a carbon-rich product with low volatile content becomes more effective as torrefaction temperature rises. Lignin maintains higher thermal stability than wood and straw due to its different chemical compositions, which determine the changing levels of magnitude among feedstocks. Basically, temperature directly affects the breakdown of hemicellulose, cellulose, and lignin in biomass, leading to substantial changes in its composition [19].

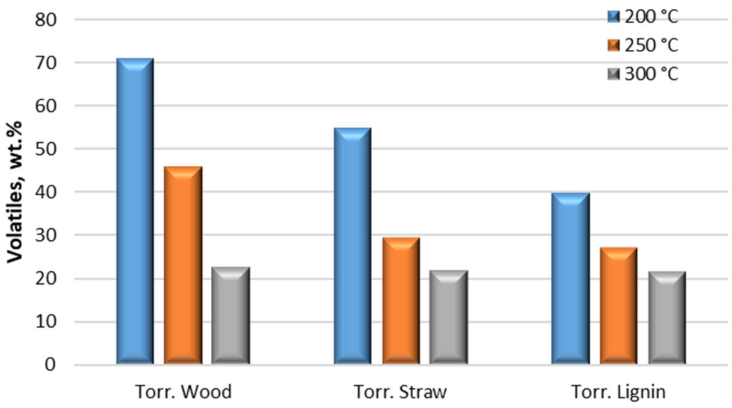


Figure 7. Temperature influence for the amount of volatiles

The extent of thermal degradation and energy densification reactions depends primarily on the specific temperature setpoint alongside residence time serving as a supporting factor for these reactions as they occur over extended period. Basically, the torrefaction temperature optimization stands as the key factor to deliver satisfactory improvements of biomass properties during energy applications, which plays a crucial role in comparison with retention time.

4.4 Analysis of liquid and gaseous phase products

As this task is dedicated for torrefied wood, straw and lignin preparation for usage in EFG, at the same time, based on the working package description, liquid and gaseous products formation must be evaluated. These products could be used as an energy carriers, or in different industries, like petrochemical or different types of chemical products or solvents production [20]. For that reason, it is imporant to analyse sub-products composition, to evaluate it‘s applicability.

The liquid phase products are mainly composed of organic acids, aldehydes, ketones, furans, and phenolic compounds, which are formed primarily from the breakdown of cellulose, hemicellulose, and lignin [21]. These compounds could be condensed and recovered for the further use in various fields. The gaseous phase, mainly composed of CO_2, CO, H_2O vapour, methane, and light hydrocarbons, results from decarboxylation, decarbonylation, and dehydration reactions [22]. In our case, during the whole procedure, the constant N_2 flow was used to create an inert ambient, with a value of 8 l/min. For that reason, the gaseous products composition is presented without an N_2, and the results are depicted in Table 4.4.

Table 4.4. *Torrefied biomass gaseous phase products composition*

Compound	**Torrefied wood**	**Torrefied straw**	**Torrefied lignin**
O_2, vol%	0.00	0.00	0.00
CH_4, vol%	4.06	3.57	3.85
CO_2, vol%	59.28	57.04	71.15
CO, vol%	32.07	31.37	19.62
C_2H_6, vol%	4.06	7.13	5.00
C_2H_2, vol%	0.00	0.00	0.00
C_3H_8, vol%	0.32	0.71	0.38
H_2, vol%	0.20	0.18	0.00

The torrefaction process gas output analysis of wood, straw and lignin demonstrated the influence of biomass raw materials on gas emissions during torrefaction. The absence of oxygen (O_2) throughout the analysis of each biomass demonstrates proper operation in oxygen-free conditions that matches torrefaction requirements to maintain inertness [22]. During the process carbon dioxide (CO_2) and carbon monoxide (CO) appear as the main gas products through decarboxylation and decarbonylation reactions [23]. Torrefying lignin produces high concentrations of carbon dioxide (71.15%) compared to wood (59.28%) and straw (57.04%) because the thermal breakdown of aromatic structures with functional groups that exist in lignin causes its degradation [24]. The greater content of carbon monoxide in wood and straw (32.07% and 31.37% respectively) as compared to lignin (19.62%) exists because hemicellulose and cellulose from wood and straw break down easily [25].

Gaseous products from straw contain the highest concentrations of ethane (C_2H_6) at 7.13% while the wood and lignin show it's ranging from 3.57% to 4.06%. The unique carbohydrate arrangement within straw makes it possible that it will generate larger quantities of light hydrocarbons [20]. All samples contain small amounts of propane (C_3H_8) while absence of acetylene (C_2H_2) demonstrates that extreme conditions of high temperature cracking or pyrolysis did not occur. The analysis shows that wood and straw contain approximately 0.20% and 0.18% hydrogen (H_2), respectively, yet lignin does not contain any trace amounts of hydrogen. Torrefaction temperatures expose lignin less frequently to dehydrogenation reactions compared to feedstocks containing higher amounts of carbohydrates [26]. Gas production quantity and breakdown pattern is primarily affected by the chemical make-up of the feedstock materials [27]. Lignin produces higher levels of CO_2 together with reduced CO and H_2 amounts compared to wood and straw which generate elevated amounts of CO and hydrocarbon gases according to the analytical results.

The liquid products composition after the torrefaction procedure analysis was maintained by the GC/Ms. These products were formed by the hemicellulose and cellulose decomposition through thermal processes together with the minor decomposition. An analysis of these complex organic mixtures containing oxygenated compounds remains essential for understanding reaction pathways while optimizing conditions and exploring possible valorization of these mixtures. The chromatograms are depicted in Figure 8.

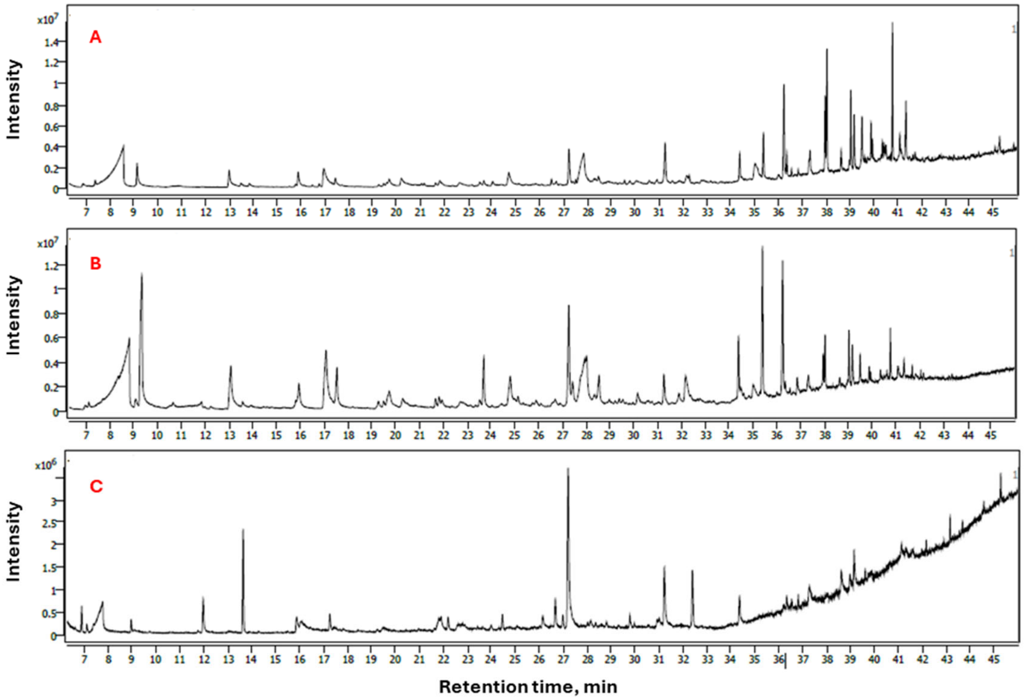


Figure 8. Chromatographic curves of liquid products from torrefaction. A – wood biomass, B – Straw biomass, C- Lignin biomass

The results demonstrate, that during the torrefaction procedure of wood along with straw and lignin produces different chemical combinations of liquid products which represent original biomass characteristics [28]. The liquid products recovered from the torrefied wood contain an equal proportion of aliphatic and aromatic chemicals between its compounds. The major component of the liquid product is acetic acid (14.3%) which provides evidence of hemicellulose breakdown [29]. Complex mechanisms are present in the rearrangement reactions because cyclopropyl carbinol (9.95%) was recovered alongside several phenolic products like phenol-2-methoxy (3.61%), phenol-2-methoxy-4-methyl (4.76%), phenol-2,6-dimethoxy (7.57%) and phenol-2-methoxy-4-(1-propenyl) (7.59%) that stem from lignin degradation [30]. The detection of 2-furanmethanol at 5.17% indicates that cellulose and hemicellulose undergo breaking down through furanic mechanisms [31]. Compounds comparison between all investigated samples are presented in Table 4.5.

Table 4.5. *Summary of key compounds by feedstock*

Compound	Wood	Straw	Lignin
Acetic acid, (%)	14.30	4.46	10.10
2-Propanone 1-hydroxy-, (%)	3.00	13.26	4.55
Phenol 2-methoxy, (%)	3.61	7.51	34.76
Cyclopropyl carbinol, (%)	9.95	–	–
2-Furanmethanol, (%)	5.17	8.70	–
Disulfide/tetrasulfide dimethyl, (%)	–	–	5.62 / 8.42
Propane 2,2-methylenebis(oxy), (%)	–	–	12.53

The liquid products obtained from torrefied straw contain greater amounts of smaller oxygenated compounds than the other products. The major product among the identified chemicals is 2-propanone 1-hydroxy- (13.26%) followed by pentanal (12.61%) and 1-hydroxy-2-butanone (5.61%). These chemical compounds are typical degradation products of carbohydrates [32]. The decomposition of polysaccharides is indicated by detectable furanic compounds particularly 2-furanmethanol (8.7%) and 3-furaldehyde (2.97%). The phenolic compounds present in the liquid include phenol-2-methoxy (7.51%) and 2-methoxy-4-vinylphenol (7.79%) but they remain less prevalent than wood and lignin compounds due to straw's lower lignin content [33].

The liquid produced during torrefaction of lignin exhibits phenolic compounds mainly consisting of aromatic molecules. Phenol-2-methoxy (34.76%) stands out as the main compound in the phenolic breakdown products of guaiacyl lignin [34]. Lignin-specific structures such as 2-methoxy-5-methylphenol and phenol 4-ethyl-2-methoxy and 2-propanone 1-(4-hydroxy-3-methoxyphenyl)- represent three of the major components in this mixture (10.91% and 4.66% and 4.55% respectively). The chemical analysis shows the presence of disulfide (5.62%) and tetrasulfide dimethyl (8.42%) compounds in addition to the main components which could stem either from sulfur-bearing raw materials or manufacturing impurities. Moreover, complex condensation reactions that take place during pyrolysis are indicated by the presence of propane 2,2-methylenebis(oxy) at 12.53% [35].

5 Conclusions and recommendations

This deliverable (D3.2) provides a comprehensive overview of torrefied biomass production by two different types of reactors (load batch and screw-type), and the main parameters – temperature and retention time – influence for the received products quality. Moreover, this biomass further will be send to test it in the EFG for syngas production. Torrefied biomass preparation makes the biomass better through decreased moisture content along with lower volatile matter content together with higher energy density properties. Biomass is used in a thermal processing under inert environments at 200–350 °C, to reach partial devolatilization which in turn makes it more suitable for grinding and storage and increases hydrophobicity. Multiple process parameters determine both the composition and amounts of solid, liquid, and gaseous products that result from the thermal treatment process. Knowledge about how to optimize by-products produced in liquid and gaseous phases matters for achieving complete energy recovery while reduced environmental emissions and enabling torrefied biomass applications in solid biofuels and subsequent thermochemical conversion processes.

As it was investigated, temperature plays a crucial role in the composition of torrefied biomass and fixed carbon content. Increased temperature significantly increases the amount of fixed carbon, decreasing the number of volatiles. However, the retention time did not affect the procedure as temperature, and its influence was negligible. Moreover, the load-batch reactor creates operational difficulties involving controlling the thermal energy output. The ongoing torrefaction reaction produces heat inside the system, which results in temperature elevation because this process is exothermic by nature. Increased temperature forces the formation of char, leading to the removal of the most significant part of the volatile. Nevertheless, this effect was removed when working with the screw-type reactor because the biomass inside the reactor always moves around, not creating the batch in one or another hot spot. For that reason, the heat flux between the particles was decreased, avoiding unfavourable increases in temperature.

6 Literature

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HISTORY OF CHANGES		
VERSION	PUBLICATION DATE	CHANGE
1.0	10.05.2025	Initial version
1.1	15.05.2025	Revised version
2.0	19.05.2025	The first version was sent to all partners
2.1	23.05.2025	Final day for partners comments
2.2	23.05.2025	Comments from partners have been taken into account
3.0	26.05.2025	The final version for submission