

D2.1. Physicochemical sheet for the biomass feedstock

FLEXBY

FLEXIBLE AND ADVANCED BIOFUEL TECHNOLOGY THROUGH AN INNOVATIVE MICROWAVE PYROLYSIS & HYDROGEN-FREE HYDRODEOXYGENATION PROCESS

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EXECUTIVE SUMMARY

This document corresponds to the technical report “*Physicochemical sheet for the biomass feedstock*”. (Deliverable 2.1).

The report contains the INCAR-CSIC results of the exhaustive analysis of six samples (bio-wastes from industrial wastewaters) considered as possible raw materials for the subsequent development of this project. Moreover, the overall information gathered in this report will be used to assess the experimental conditions to be defined in subsequent FLEXBY tasks.

Two of the samples were supplied by A4F, a consortium partner in charge of sample delivery. Given the intrinsic fossil nature of the oily sludge samples from GALP, these residues were replaced by the following bio-based alternatives: 2 samples from the macroalgae industry and 2 samples from the dairy industry. These samples were distributed directly by INCAR-CSIC.

The samples were fully characterized in terms of water content, ultimate analysis (C, H, N, S, O), ash content, volatile matter content, chlorine and potassium content, heating value, ash melting point, thermogravimetric analysis and dielectric properties. The volatile content and its thermogravimetric analysis were also analyzed to fully characterize the feedstocks and determine the temperatures at which the thermal process should be carried out.

The second section of the report (2. Experimental section) specifies the ISO/EN or ASTM standards used in each test, while section five (5. References) lists these standards.

The results provide evidence surrounding the suitability of the samples to be used as raw materials in this project. Four of the wastes (Feedstock 1, 2, 5, 6) contained high carbon content in the range of 40-54%, low-moderate ash content in the range of 13-24% and high volatile content in the range of 60-80%. The other two wastes (Feedstock 3 & 4) had a chemical composition which makes them less suitable for the project purpose. This was due to their low carbon content (18% and 28%) and their high ash content (60% and 41%). The results of the thermogravimetric testing on the wastes indicated that the optimal temperature for microwave pyrolysis should be 600°C or above. In terms of dielectric properties, all samples showed effective absorption of microwave energy, suggesting that the samples are suitable for use in microwave pyrolysis.

1. INTRODUCTION

1.1 DESCRIPTION OF THE DOCUMENT AND PURSUE

This report, Deliverable 2.1, represents the "Physicochemical Sheet for Biomass Feedstock" and has been submitted in month 6 of the FLEXBY project. The report aims to evaluate the suitability of selected raw materials used as feedstock for the project. This report outlines the results of the physicochemical characterization of these samples, including their dielectric properties, following ISO/EN or ASTM standards.

The physicochemical characterization conducted at CSIC facilities included analyses of water content, ultimate composition (C, H, N, S, O), ash and volatile matter content, chlorine and potassium levels, heating value, ash melting point, and thermogravimetric properties. FRIMA analyzed the dielectric properties—specifically, the dielectric constant and dissipation factor across different frequencies and temperatures—of the biomass waste. These parameters are critical in selecting the most suitable samples for the subsequent stages of the project.

1.2 WPS AND TASKS RELATED WITH THE DELIVERABLE

Deliverable 2.1 is part of the Work package 2: Flexby requirements and will report on the following tasks:

2.1: "Oily sludge physico-chemical characterization"

2.2 "Identification and characterization of microalgae"

1.3 ROLES & RESPONSABILITIES

The roles and responsibilities regarding the physico-chemical analysis in the Flexby project are clearly defined and distributed among the consortium members to ensure effective execution and maximum impact:

CSIC: is responsible for the physico-chemical analysis of the samples and also provides support as the main contact to the industrial sludge providers (Feedstock 3,4,5 & 6).

FRIMA: is responsible for testing the dielectric properties of the feedstocks.

A4F: is a feedstock provider for microalgae samples.

1.4 DEVIATIONS

During the execution of the activity related to this deliverable, the following deviation was reported: one of the feedstock suppliers, GALP, responsible for providing three samples of oily sludge, alerted that they was unable to supply the material because their feedstock would compromise the bio-based nature of the final biofuel product. Consequently, during the first General Assembly in May 2024, the consortium confirmed the need to seek industrial suppliers that could provide a similar oily sludge with optimal quality for biofuel production in line with the project's technologies.

In this regard, CSIC made contact with three industries: one macroalgae processing industry and two dairy industries. These companies expressed interest in participating as industrial waste suppliers for the FLEXBY project.

In addition, the granulometry of the waste (included as activity in Task 2.1) was not studied because the samples were in the form of a “paste” and it was not considered critical for the characterisation of the waste, unlike the volatile content and the thermogravimetric analysis. Additionally, volatile content and thermogravimetric analysis were studied to improve the quality of the physico-chemical characterisation.

2. EXPERIMENTAL SECTION

2.1 Raw Materials

For the characterization of the samples, six different types of feedstocks were selected: Feedstock1 and Feedstock2 are microalgae cultivated in urban wastewater; Feedstock3 and Feedstock4 are sludge from the wastewater treatment of the macroalgae industry, and Feedstock5 and Feedstock6 come from the dairy industry.

In order to maintain confidentiality until the publication of results, the origin and names of the samples, including the companies, will not be disclosed in this deliverable.

2.2. Sample drying

Before characterization, the six samples supplied were dried in a forced-air oven at 40°C and for different times, depending on the amount of sample used. The drying yield was calculated as the sample weight difference before and after the treatment. For clarity, all samples keep their name after drying.

2.3. Sample characterization

2.3.1. Proximate Analysis. Ultimate Analysis.

The Proximate Analysis includes the determination of the content of moisture, ash, volatile matter and fixed carbon. Moisture, ash and volatile matter content were determined on a TGA 701 LECO (LECO Corporation, Groveport, Ohio, United States). The test is performed according to the ASTM D7582-10 standard. The volatile matter content was also analysed, although this was not originally a requirement of the project. This provides valuable information about the material to be pyrolyzed.

The ultimate analysis provides the percentage content of the elements carbon, hydrogen, nitrogen, sulphur and oxygen present in the sample. Determination of carbon (C), hydrogen (H) and nitrogen (N) content was conducted using a LECO CHN-2000 equipment instrument (LECO Corporation, Groveport, Ohio, United States). The test is performed according to the ASTM D5373. Sulphur (S) content was quantified on a LECO S632 instrument (LECO Corporation, Groveport, Ohio, United States). The test is performed according to the ASTM D4239. The oxygen (O) content was calculated by difference.

2.3.2. Heating Value

The calorific value of a sample is defined as the amount of heat released when a given quantity of the sample is burnt under standardised conditions. The higher heating value (HHV) represents the net heat produced in the combustion reaction, while the lower heating value (LHV) corresponds to the actual usable heat.

The HHV of the biomass residues was determined in an IKA C4000 adiabatic calorimeter. The test was performed according to ASTM D5865/D5865M-19.

Equation 1 shows the correlation between HHV and LHV, considering the latent heat of evaporation of water at 25 °C, which is 49.24 kcal/kg.

$$\text{LHV} = \text{HHV} - 49.24 \text{ H} \quad \text{eq. (1)}$$

where, H is the hydrogen content expressed as a percentage on the dry basis.

2.3.3. Chlorine determination

Total chlorine is determined according to the ASTM D4208-19 standard procedure. In brief, this method follows combusting a weighed sample in an oxygen bomb with dilute base adsorbing the chlorine vapors. The bomb is rinsed into a beaker with water and following the addition of an ionic strength adjuster, the chlorine is determined by ion-selective electrode.

2.3.4. Potassium content in bio-waste by XR-fluorescence analysis of mineral matter.

The potassium content of the biomass waste was calculated from the results obtained from the inorganic composition of the biomass waste ashes analyzed by XRF. The analysis was performed on a Bruker S8 X-ray fluorescence spectrometer equipped with a Rhodium (Rh) tube as an X-ray source. Sample preparation for XRF analysis was carried out using the fused bead method. The XRF analysis was conducted in accordance with the ISO 12677:2003 standard.

2.3.5. Ash melting point

An understanding of the ash melting characteristics of biowastes provides insight into the potential behavior of the inorganic components of these residues when subjected to increasing temperatures. In general, a high ash melting temperature is preferred to avoid the deposition of sticky ash in the furnace. The ash fusibility test is carried out in an oxidizing atmosphere and the test is performed on automatic equipment LECO AF700 (LECO Corporation, Groveport, Ohio, United States). The test is performed according to the ASTM D1857/D1857M-18.

Sample preparation: a pyramidal specimen is prepared from the ash of the sample. Fusibility test: the pyramidal specimens are heated in a furnace under standardised conditions (ASTM D1857/D1857M-18) and the temperatures at which the specimen undergoes changes are studied:

- IT = Initial Deformation Temperature
- ST = Softening Temperature
- HT = Hemispherical Temperature
- FT = Fluid temperature

2.3.6. Thermogravimetric analysis

Thermogravimetric analysis was carried out using a TGA-Q5000IR thermobalance (TA Instruments, New Castle, DE, EE. UU.).

The instrument was earlier calibrated using a known standard to guarantee the correct functioning of the thermobalance. A small representative sample (about 10-20 mg) was placed in a platinum crucible and heated under a N₂ atmosphere (25 ml min⁻¹). A heating rate of 5 °C·min⁻¹ was applied until a final temperature of 900 °C was achieved. Through this procedure, thermogravimetric profiles (TG) were obtained representing the weight loss with respect to temperature. To identify the different stages, it is advisable to derive these TG profiles (DTG profiles).

2.3.7. Dielectric properties

In this study, the dielectric properties of the 6 samples provided were measured using a high-temperature dielectric characterization system. The primary focus was on determining the relative permittivity (ϵ') of the sample at a microwave frequency of 2.45 GHz under varying temperature conditions, aiming to assess the effect of moisture and thermal treatment on the dielectric response of the macroalgae.

1. Sample Preparation

The sample was prepared by manually compressing it into a cylindrical shape within a sample holder to minimize air pockets, thus ensuring better contact and reducing measurement artifacts. The resulting cylinder had a diameter of 6.86 mm and a height of 6.39 mm. This sample was positioned in a cylindrical sample holder designed to maintain sample integrity and prevent significant morphological changes during temperature treatments.

2. Dielectric Measurement Conditions

Dielectric measurements were conducted using a high-temperature dielectric characterization machine capable of measuring material properties under controlled temperature conditions. The dielectric properties were measured in terms of relative permittivity (ϵ') at a frequency of 2.45 GHz, which is relevant for applications in microwave processing and heating.

Measurements were carried out under the following temperature conditions:

3. Initial Temperature Steps: The sample was measured initially at room temperature, followed by sequential heating to 50°C, 100°C, and 150°C. These temperature steps were chosen to observe the dielectric response of the macroalgae powder as a function of thermal exposure, likely linked to variations in water content and structural changes at higher temperatures.

4. Residual Water Content Measurement: After the initial measurements, the sample was left to cool, and a second set of measurements was conducted the following day. These measurements, performed at room temperature and then at 100°C, were aimed at assessing the dielectric properties after potential residual water content had been removed from the sample.

5. Dielectric Characterization Equipment and Procedure

The dielectric measurements were carried out using a high-temperature dielectric characterization system, which provides high precision for both permittivity and loss factor at microwave frequencies. The system's design ensures stable temperature control, which is essential to achieving accurate measurements across the selected temperature range. Temperature stability and accurate frequency settings at 2.45 GHz allowed for consistent measurements across different thermal conditions.

Each measurement was recorded after allowing adequate time for the sample to reach thermal equilibrium at the target temperature to prevent transient effects from influencing the permittivity values. The dielectric response at each temperature was obtained in three replicates to ensure repeatability and reliability of the results.

By following this methodology, the dielectric properties of the samples were comprehensively analysed, providing insight into the impact of temperature and moisture content on the material's relative permittivity. This methodological approach provides a basis for understanding the potential applications of the 6 samples in microwave processing for other dielectric-sensitive applications.

3. RESULTS

3.1 Sample reception

As described in the previous section, 6 samples were analyzed and are presented in this report:

The first two samples (Feedstock1 and Feedstock2) were supplied by A4F. These are two microalgae-based wastewater residues and were received as humid residues in 50 mL Eppendorf tubes (Figure 3.1).



Figure 3.1. Microalgae-based wastewater residues (Feedstock1 and Feedstock2).

The initial quantity of microalgae samples was insufficient to complete all the planned analyses. During October 2024, A4F supplied additional quantity of Feedstock2 sample.



Figure 3.2. Microalgae-based wastewater residues (Feedstock2).

Two macroalgae processing industry samples were provided to CSIC named as Feedstock3 and Feedstock4. These samples correspond to industrial sludges obtained after the first and second cycles of their wastewater recovery (Figure 3.3).

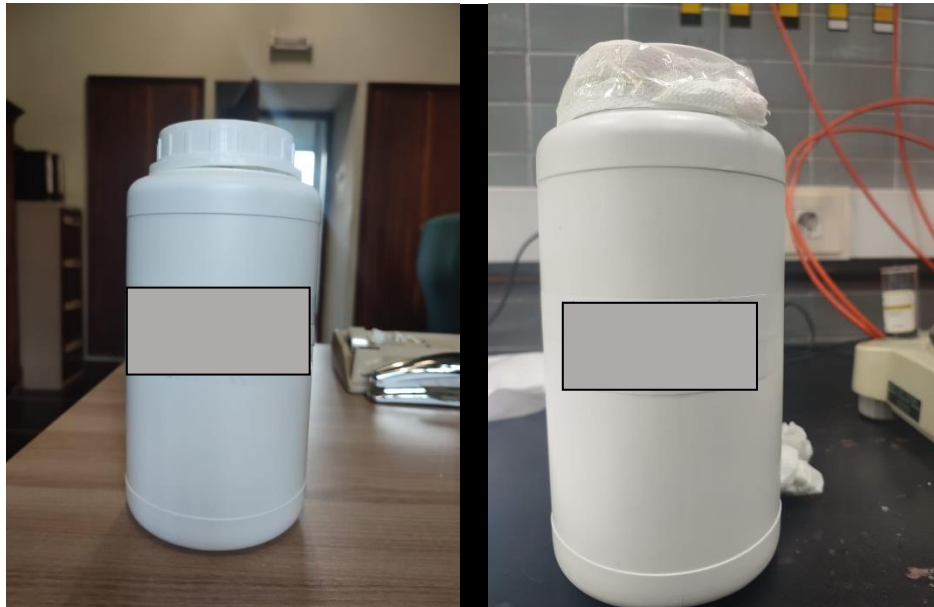


Figure 3.3. Wastewater residues, obtained in two different centrifugation cycles, supplied by the macroalgae industry (Feedstock3 and Feedstock4).

Two oily sludges from two different dairy industries were supplied to CSIC, named as Feedstock5 & 6 (Figure 3.4 and Figure 3.5, respectively).

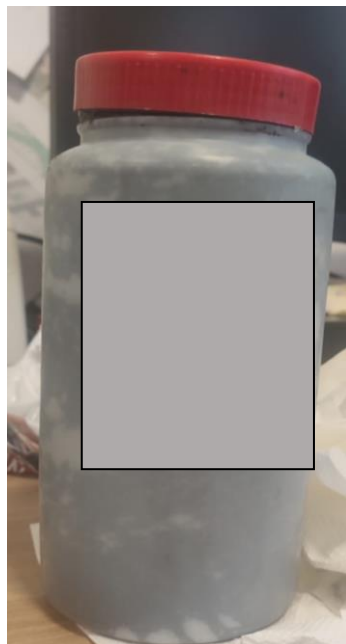


Figure 3.4. Dairy-based oily-sludge obtained from dairy industry1 (Feedstock5).



Figure 3.5. Dairy-based oily-sludge obtained from dairy industry 2 (Feedstock6)

All samples were properly stored at 5°C.

3.2 Sample drying

All samples were supplied as centrifuged wastewater residues. Prior to any characterisation, all samples were dried in a forced-air stove at 40°C, for a variable period depending on weight, to remove most of the water content. This was a prerequisite for processing the samples in the subsequent steps (Figure 3.6). The stove selected to dry the Feedstock3, Feedstock4, Feedstock5 and Feedstock6 was located outdoors to easily manage the samples.



Figure 3.6. Residue (left) as received and (right) in the stove.

All samples were weighed before and after treatment to calculate the drying yield. The results obtained are summarized in Table 1.

It is generally observed that the dry sample yield is around 14-24%, which indicates that a significant amount of water (86-76%) remains after the industrial centrifugation processes which were used to obtain them.

Table 1: Experimental conditions of sample drying and dry sample yields

Biomass waste	Drying temperature (°C)	Drying time (h)	Amount of sample to be dried (g)	Dry sample yield (wt.%)
Feedstock1	40	17 h	45	14.1
Feedstock2	40	17 h	50	15.7
Feedstock3	40	48 h	934	20.7
Feedstock4	40	48 h	684	14.0
Feedstock5	40	48 h	1115	15.5
Feedstock6	40	48 h	635	24.1

After drying, the samples were crushed to reach an appropriate size for further analysis.

3.3 Proximate and Ultimate analysis characterization

The 6 samples were characterized by means of proximate and ultimate analysis, and the results obtained are summarized in Table 2.

Table 2: Proximate and ultimate analysis of the feedstocks

Biomass waste	Moisture (%)	VM (%) ^a	Ash (%) ^a	C (%) ^a	H (%) ^a	N (%) ^a	S (%) ^a	O (%) ^{a, b}
Feedstock1	10.93	62.7*	24.43	40.56	5.80	5.79	0.35	23.07
Feedstock2	9.84 6.59**	62.98**	22.26 34.00**	41.73	6.19	4.10	0.25	25.47
Feedstock3	17.48	36.19	60.22	18.78	2.36	3.58	1.06	14.00
Feedstock4	14.76	51.25	41.07	28.58	3.72	5.47	1.00	20.16
Feedstock5	8.91	72.29	22.56	44.20	6.18	5.50	0.55	21.01
Feedstock6	7.46	79.80	13.35	54.31	8.45	5.88	0.38	17.63

^a dry basis

^b calculated by difference

* Due to the lack of Feedstock1, its volatile matter content was estimated from the pyrolysis curve.

** Results of sample Feedstock2 received in October 2024, equivalent to Feedstock2 received in July 2024.

One of the most notable results from these analyses is the ash content of the samples. Since ash represents the mineral matter (non-carbonaceous fraction) within these residues, minimizing this parameter is desirable for the project's objective, which focuses on upgrading the carbonaceous fraction of bio-wastes. As shown in Table 2, the highest ash content was found in Feedstock3 (60.22 wt%) and Feedstock4 (41.07 wt%), both derived from macroalgae wastes. This high ash content primarily reflects sand residues separated from raw algae during early industrial processing stages. In contrast, Feedstock6 exhibited the lowest ash content (13.35 wt%) and the highest volatile matter content (79.8%), making it particularly valuable for the project. Additionally, the moderate ash content (22-24%) of Feedstocks 1, 2, and 5 indicates their suitability as feedstocks for this project.

Volatile matter content was also analysed, although it was not originally a project requirement. This analysis provides valuable insights into the material's pyrolysis potential. Feedstocks 1, 2, 5, and 6 show high volatile matter content (60-80%), which aligns very well with the project's objectives. Feedstock3 and Feedstock4, however, have lower volatile contents (36% and 51%, respectively).

3.4 Higher heating value (HHV) and lower heating value (LHV)

The calorific value (HHV and LHV) of the 6 samples are included in Table 3. The heating value is expressed in Kcal/kg and MJ/kg.

Table 3: Calorific value of the biomass waste

Biomass waste	HHV (Kcal/Kg)	HHV (MJ/Kg)	LHV (Kcal/Kg)	LHV (MJ/Kg)
Feedstock1	4250	17.8	3966	16.6
Feedstock2	4434	18.6	4130	17.3
Feedstock3	1737	7.3	1622	6.8
Feedstock4	2966	12.4	2784	11.6
Feedstock5	4681	19.6	4378	18.3
Feedstock6	6120	25.6	5706	23.9

As expected, Feedstock3 had the lowest HHV (7.3 MJ/kg) due to its high ash content (60.22%) and low carbon content (18.78%) (see Table 2). Feedstock4, with a high ash content (41.07%), presented a low HHV (12.4 MJ/kg), although slightly higher than that of the Feedstock3. Feedstock6 had the highest HHV (25.6 MJ/kg) in line with its low ash and high carbon content. Feedstock1, Feedstock2 and Feedstock5 presented HHV values between 17 and 20 MJ/kg.

3.5 Determination of the chlorine content

The chlorine content in the biomass waste was determined by means of the standard ASTM D4208-19 procedure. Table 4 shows the chlorine content of the residues.

Table 4: Chlorine content in the biomass waste

Biomass waste	% Cl
Feedstock1	*
Feedstock2	0.11**
Feedstock3	0.14
Feedstock4	0.22
Feedstock5	0.052
Feedstock6	0.015

**This analysis could not be performed due to lack of Feedstock1.
**This analysis was performed with Feedstock2 received in October 2024*

The presence of chlorine compounds in the waste can lead to corrosion of the pyrolysis equipment. The level of corrosion risk can be preliminarily assessed by determining the chlorine content of the biomass waste. It is estimated that there is no corrosion risk at $Cl < 0.02\%$ (Pelucchi et al., 2015).

The highest levels of chlorine content were presented in Feedstock3 and Feedstock 4 (0.14% and 0.22%). These levels indicated a risk of corrosion. Feedstock6 had the lowest chlorine content (0.015%), which is below the estimated threshold for corrosion risk.

3.6 Potassium content by XR-fluorescence analysis of mineral matter.

The potassium content in the biomass waste is between 0.13 and 0.85% (see Table 5).

In biomass gasification processes with CO_2 , the presence of basic oxides (CaO, MgO, Fe_2O_3) or alkalis (Na_2O , K_2O) promotes the gasification rate of organic matter, increasing its reactivity (Diez and Borrego, 2013). As can be seen in Table 5, Feedstock3 and Feedstock4 had the highest potassium content and would therefore have a higher reactivity in CO_2 gasification processes.

Table 5: Potassium content in the biomass waste

Biomass waste	% K
Feedstock1	0.31
Feedstock2	0.38
Feedstock3	0.85
Feedstock4	0.84
Feedstock5	0.13
Feedstock6	0.16

3.7 Ash melting point

Table 6 shows the values of the characteristic ash fusibility test temperatures for the different feedstocks. The test was carried out in an oxidizing atmosphere.

Table 6: Characteristic ash fusibility test temperatures for the biomass waste

Biomass waste	IT(°C)	ST(°C)	HT(°C)	FT(°C)
Feedstock1	*	*	*	*
Feedstock2	>1500**	>1500**	>1500**	>1500**
Feedstock3	1205	1252	1323	1438
Feedstock4	1092	1304	1331	1349
Feedstock5	1336	1397	1412	1467
Feedstock6	>1500	>1500	>1500	>1500

*This analysis could not be performed due to lack of Feedstock1

**The sample used in this analysis was Feedstock2 received in October 2024.

Initial deformation temperature (IT), is the temperature at which the first rounding of the pyramid apex occurs.

Softening temperature (ST), is the temperature at which the pyramid has fused down to a hemispherical lump at which point the height is one-half the width of the base.

Hemispherical temperature (HT), is the temperature at which the pyramid has melted to form a hemispherical lump in which the height is half the width of the base.

Fluid temperature (FT), is the temperature at which the fused mass has spread out in a nearly flat layer with a maximum height of 1.6 mm.

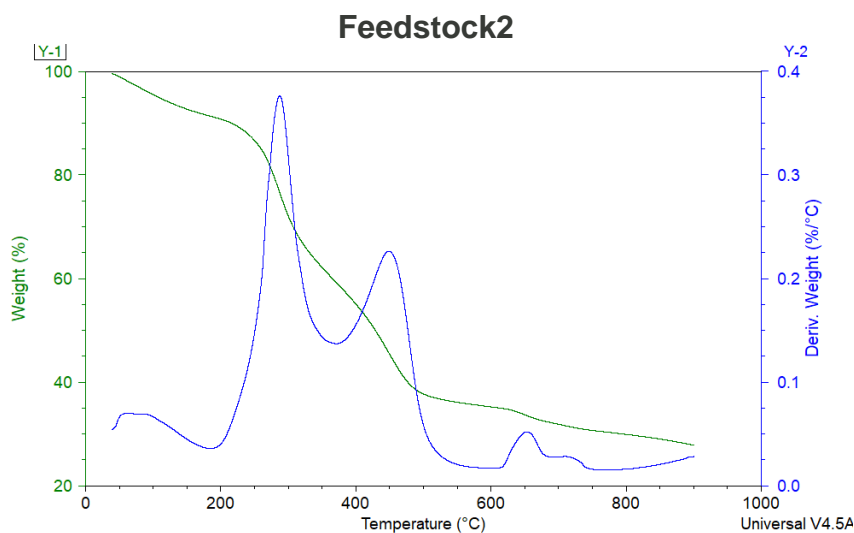
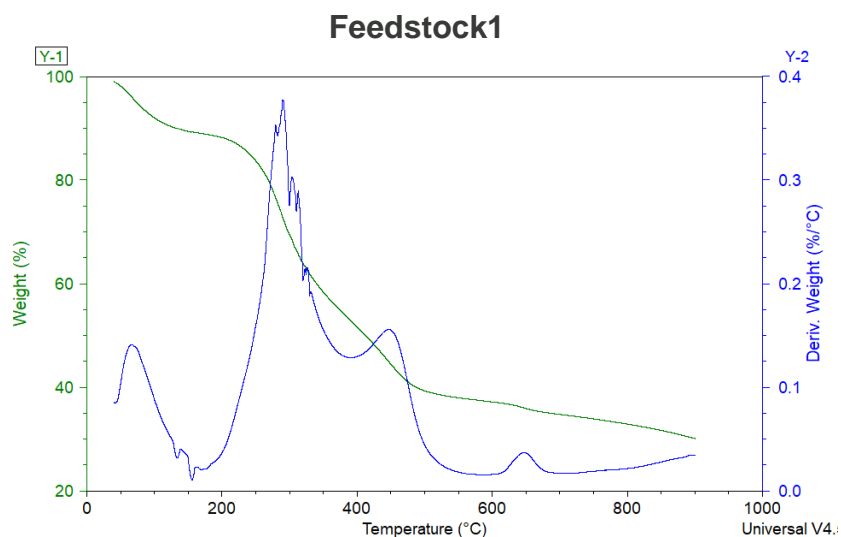
The primary factors affecting the fusibility of ash are its chemical composition and the surrounding environmental atmosphere (Che, 2008). In general, the ash fusibility of fuels is considered good when the ash melts above 1500 °C, normal when it melts between 1500 and 1200 °C, and poor when it melts below 1200 °C. However, the acceptability of ash fusibility ultimately depends on the maximum temperatures to which the ash is exposed and the way in which the fuel is used.

This analysis could not be performed on Feedstock1 due to a lack of sample. Feedstock3 and Feedstock4 ashes showed the lowest initial deformation temperature. Feedstock 2 and Feedstock 6 ashes presented initial deformation temperature, softening temperature, hemispherical temperature and fluid temperature above 1500 °C, indicating that these ashes can be classified as refractory ashes.

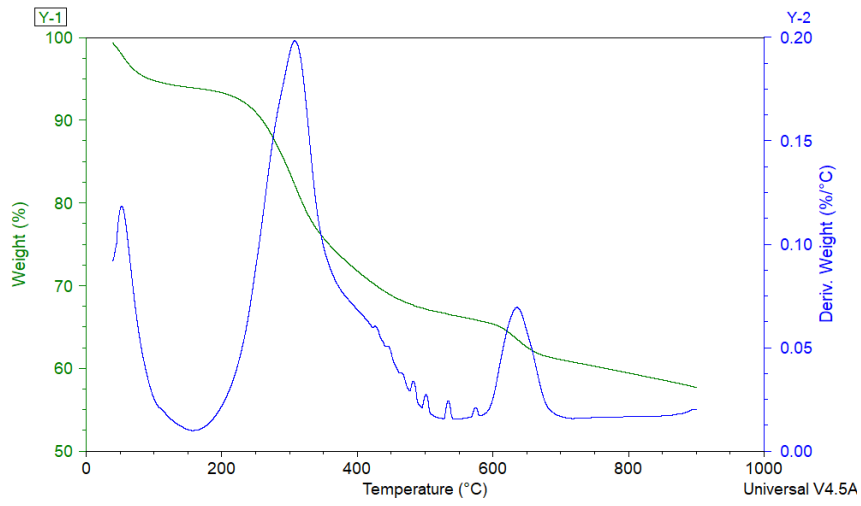
3.8 Thermogravimetric analysis

The thermogravimetric analysis was conducted to determine the characteristics of the volatile matter present in the samples. Although this analysis was not specified in Task 2.1, it was deemed necessary following prior discussions with the technical partners due to the importance of identifying the temperatures at which volatiles are released from the samples. This provides valuable insight into the material to be pyrolysed.

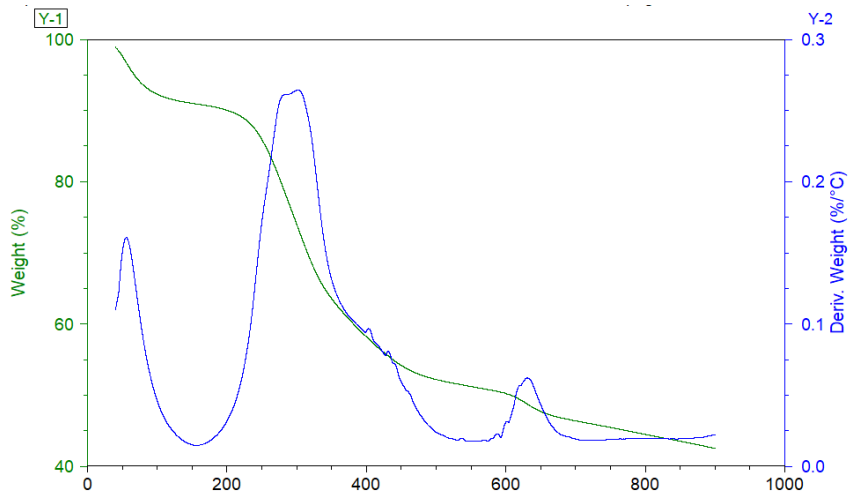
The thermal stability of the samples was determined by means of thermogravimetric (TG) analysis. The TG curves obtained for all samples analysed showed a small weight loss up to 100 °C, which was mainly due to moisture loss. The greatest weight loss occurs between 100 and approximately 600 °C for all bio-waste samples. Between 600 and approximately 900 °C, a further minor loss is observed. Based on the thermograms, the temperature at which pyrolysis of feedstocks should be carried out is 600 °C, and slightly higher temperatures (700, 800 °C) need to be tested. In the DTG curves, the maximum rate of change presented by biomass residues in pyrolysis can be identified, which is in the range of 290-310 °C. The TG and DTG curves of the different feedstocks are presented below, Fig. 3.7.



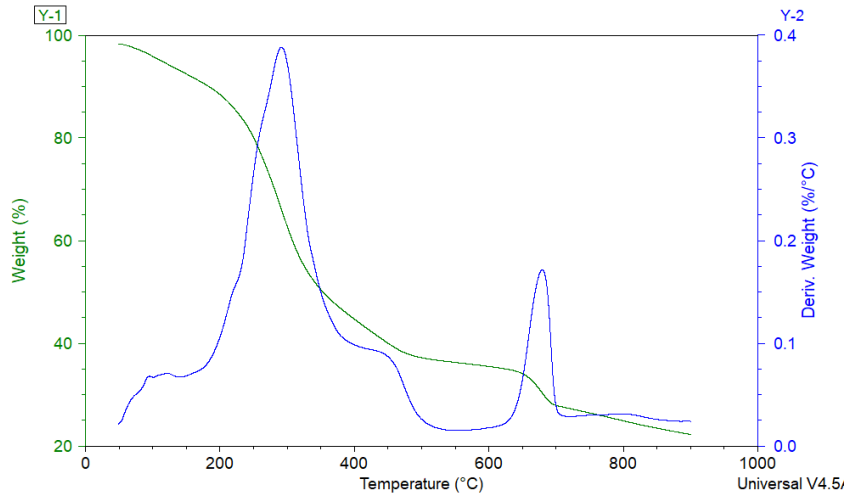
Feedstock3



Feedstock4



Feedstock5



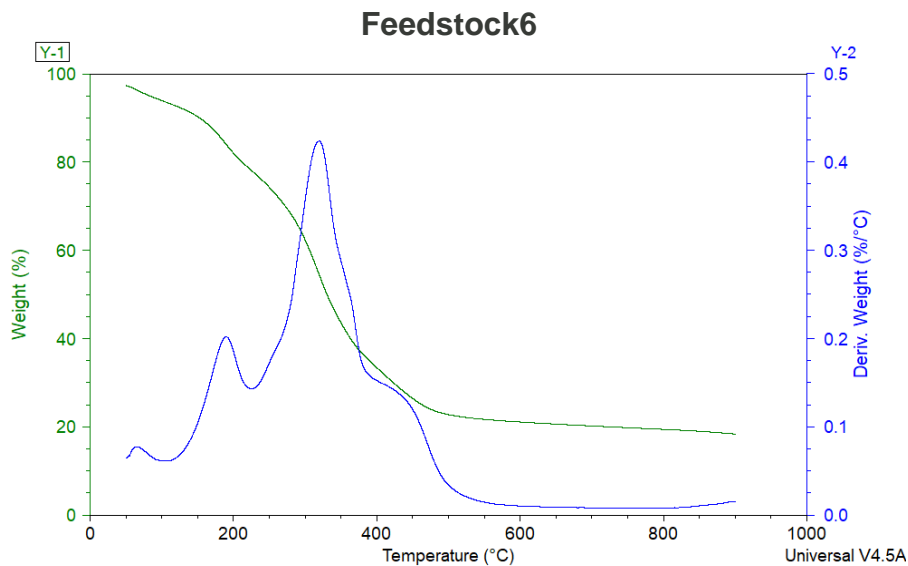


Figure 3.7. TG and DTG curves of the biomass waste

3.9 Dielectric properties

The initial measurements (Cycle 1) of the macroalgae powder samples revealed a relatively high relative permittivity (ϵ') up to a temperature of 100°C, primarily due to the presence of residual water content. This water influenced the dielectric properties, as moisture within the samples increased permittivity at lower temperatures. At temperatures above 100°C, most of the residual water evaporated, resulting in a decrease in permittivity values. Consequently, the second measurement cycle, conducted after residual water had largely evaporated, exhibited lower permittivity values, providing a more accurate representation of the samples in a dry state.

Water content varied between the different feedstocks, impacting their dielectric behaviour. Specifically, Feedstock3 and Feedstock4 showed the highest initial permittivity values due to higher water content, whereas Feedstock1 and Feedstock2 samples had the lowest moisture levels, resulting in correspondingly lower permittivity values. Above 100°C, the samples exhibited closer permittivity values, indicating that the impact of water content diminished as moisture evaporated at higher temperatures.

Dielectric Data Tables

The following tables summarize the relative permittivity (ϵ'), dielectric loss factor (ϵ''), and loss tangent ($\tan(\delta)$) for samples across two cycles at each measured temperature. The temperature-dependent dielectric properties for each sample at a frequency of 2.45 GHz are displayed below:

Table 7: Dielectric properties for the samples

ϵ' at 2.45 GHz	T (°C)	Feedstock1 (L2024062001)	Feedstock2 (L2024062002)	Feedstock3	Feedstock4	Feedstock5	Feedstock6
Cycle 1	20	1.663	1.886	4.998	3.589	2.589	2.389
	50	1.759	1.946	5.500	4.082	2.804	2.569
	100	1.864	2.037	5.013	4.506	3.318	3.068
	150	1.726	1.923	1.967	2.182	2.446	2.233
Cycle 2	20	1.650	1.838	1.946	2.099	2.261	2.119
	100	1.739	1.937	2.050	2.204	2.395	2.232

ϵ'' at 2.45 GHz	T (°C)	Feedstock1 (L2024062001)	Feedstock2 (L2024062002)	Feedstock3	Feedstock4	Feedstock5	Feedstock6
Cycle 1	20	0.0454	0.0302	1.3970	0.5768	0.1106	0.0903
	50	0.0726	0.0452	1.7550	0.6981	0.1930	0.1560
	100	0.0855	0.0639	1.7323	0.9640	0.4058	0.3126
	150	0.0403	0.0323	0.0395	0.0587	0.0905	0.0624
Cycle 2	20	0.0158	0.0127	0.0216	0.232	0.0240	0.0225
	100	0.0339	0.0272	0.0440	0.0462	0.0593	0.0502

$\tan(\delta)$ at 2.45 GHz	T (°C)	Feedstock1 (L2024062001)	Feedstock2 (L2024062002)	Feedstock3	Feedstock4	Feedstock5	Feedstock6
Cycle 1	20	0.0273	0.0160	0.2795	0.1607	0.0427	0.0378
	50	0.0413	0.0232	0.3191	0.1710	0.0688	0.0607
	100	0.0459	0.0314	0.3456	0.2139	0.1223	0.1019
	150	0.0234	0.0168	0.0201	0.0269	0.0370	0.0279
Cycle 2	20	0.0096	0.0069	0.0111	0.0111	0.0106	0.0106
	100	0.0195	0.0140	0.0215	0.0210	0.0248	0.0225

4. CONCLUSION

To ensure the successful development of the Flexby project, it was essential to identify biomass residues (Feedstocks) that align with the project's objectives. A4F, a project partner, provided two microalgae-derived samples from wastewater, while GALP, another project partner, alerted that it could not provide suitable samples for the project since the final products should have bio-based nature. Consequently, CSIC sourced four alternative industrial sludge samples from three different companies in the Asturias region of Spain. This substitution of the original three GALP residues with four new industrial residues **has been officially reported as a project deviation**. As a result, **six residues** were available for analysis: two oily sludges from the dairy industry, two macroalgae processing sludges, and two microalgae residues. **All six residues underwent a detailed characterization to assess their suitability based on the project's objectives.**

The samples arrived in paste form with high moisture content. Following the drying process, it was determined that the samples contained approximately 76-86% water, yielding 14-24% dry biomass (material to be pyrolyzed) from the initial quantity received. The analysis of ash and volatile matter content varied among residues, with a preference for those feedstocks with low ash content. Four feedstocks had an ash content of less than 24%, with one showing an ash content as low as 13%, making them particularly suitable for the project. However, Feedstocks 3 & 4 displayed high ash contents of up to 60%.

In terms of volatile matter content, four samples exhibited high levels (up to 80%), while Feedstocks 3 and 4 showed lower volatile matter levels (30-50%). Carbon content analysis revealed that four samples had satisfactory levels (40-54%), though Feedstocks 3 and 4 had notably low carbon contents of approximately 18% and 28%, respectively. Monitoring chlorine compounds in the samples was also critical, as high chlorine levels can corrode pyrolysis equipment. Feedstocks 3 and 4 exhibited elevated chlorine levels, indicating a corrosion risk. Thermogravimetric testing suggested that microwave pyrolysis should be conducted at temperatures above 600°C. The dielectric properties of the samples demonstrated effective microwave energy absorption, with loss tangent ($\tan(\delta)$) values generally around 10^{-2} , indicating suitable dielectric loss for microwave-assisted processes. Specifically, Feedstocks 3 and 4 showed the highest absorption, with $\tan(\delta)$ values reaching up to 0.3456 at 100°C in the first cycle, correlating with their higher water content. This absorption efficiency suggests that these samples are well-suited for microwave-driven pyrolysis, where efficient heating and material decomposition are essential.

Nevertheless, the chemical analysis results indicated that Feedstocks 3 and 4 are not suitable for the cost-efficient production of biofuels due to their high ash content (~60% and ~41%) and carbon content (~18% and ~28%), both below the 40% threshold. **Despite their favorable dielectric properties, it is recommended that Feedstocks 3 and 4 should not be considered as feedstock options for the project.** In contrast, the remaining biomass wastes (**Feedstock1, Feedstock2, Feedstock5, and Feedstock6**) align well with the project's goals, with Feedstock6 being the most favorable, as it exhibited the lowest ash content (~13%), the highest carbon content (~54%), and the highest volatile matter content (~80%).

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