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D3.2
Testing VINTER intermediate biofuel batches

Decentralised production of advanced biofuels for maritime shipping

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1. Introduction

1.1 Description as per Grant Agreement

This Deliverable is an analytic report on crude oil quality based on the biofuel intermediates ("bio-oil") produced in the pilot-scale VINTER unit at the Fraunhofer UMSICHT facilities in Hohenburg, Bavaria (Germany) from the **three main feedstocks**, *i.e.*, biowaste sieving material, rice husk and agave bagasse. This will be done based on 200-liter batches of biofuel. This Deliverable is related to **Task 3.3**.

- ***Task 3.3: Production of first 200 litre batches of intermediate biofuel*** *(Lead: Fraunhofer UMSICHT, Partners: DTU) [M06-M10].* This task revolves around campaigns for production of at least 200 litres of bio-crude VINTER oil for analytic validation at FRAUNHOFER as well as for testing at FVTR (*cf*. Task 3.4). To produce 200 litres of intermediate biofuel VINTER oil, campaigns with a duration of five days are necessary. This helps validate the intermediate oil qualities at pilot-scale and set a benchmark for the scaled-up version of the VINTER demo unit. FRAUNHOFER will provide at least three batches for FVTR to have more oil for testing as a fallback. The intermediate fuel production will be performed with the process parameters set in Task 3.2.

For additional context, Task 3.2 and Task 3.4, which are mentioned in Task 3.3, are also described below:

- ***Task 3.2: Process optimisation for advanced bio-oil qualities*** *(Lead: Fraunhofer UMSICHT, Partners: DTU) [M03-M06].* In this task, the process will be optimized by FRAUNHOFER in various steps, e.g., altering the process parameters to gain additional quality and yields of the bio-oil intermediate. The knowledge gathered from previous tests, done in T3.1 will be taken into account in this task. Parameters such as the specific energy demand and temperatures of heating flue gas are crucial for further scale up. As an estimation, two to four weekly trials are needed to set the parameters correctly. Tests will be done for each of the selected feedstocks to be aware of differences in the process performance. A report will be compiled prior to the production of the first 200 litre batch of intermediate biofuel.
- ***Task 3.4: Testing funnel of first VINTER intermediate biofuel batches*** *(Lead: FVTR, Partners: Fraunhofer UMSICHT) [M07-M11].* The standardised testing funnel of the first batches of VINTER intermediate biofuels will take place. In advance of the testing at FVTR, the intermediate biofuel will be analysed with standard analytics at FRAUNHOFER to gain a first glance on specifications of the VINTER oil. Therefore, specifications such as TAN (total acidic number), water content, elementary composition and the heating value of the oil will be analysed using the laboratory at FRAUNHOFER and based on ISO 8217:2017 marine fuel standards. These analytics are complementary to the ones FVTR will perform and will provide information also to the value chain simulation tool. This will be done (in part) parallel to T3.3 as the campaign duration to produce the needed 200 litres for one batch of bio-oil, one week of continuous operation on the VINTER pilot unit.

1.2 Deviations and additional information

In coordination with the Project Officer, and as a deviation from the original proposal, digestate from biowaste was prioritised as an additional project feedstock, while the experiments on rice husk were postponed. Also, as communicated to the Project Officer, a deviation was reported for the extension of Task 3.2 and, consequently, also Task 3.3 and Task 3.4, mainly due to substantial delay in the import of the agave bagasse feedstock from Mexico into Germany. This Deliverable was therefore moved for submission from M11 to M18 – still within the first Reporting Period.

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2. Executive Summary

This Deliverable reports the production and basic characterisation of bio-oil batches generated in the pilot unit at Fraunhofer UMSICHT's industrial facilities. To assess the quality and suitability of the produced bio-oils for downstream activities in SEAFAIRER, the following analytical methods were applied: **Karl Fischer titration** (water content), **CHNS** elemental analysis (bulk composition), **bomb calorimetry** (heating value), and **total acid number** (TAN) titration (acidity).

The bio-oil samples were produced by Fraunhofer UMSICHT in the pilot unit; further details on the unit configuration and operating conditions are provided in Deliverable D3.1. Bio-oil is the main product from the pilot runs and constitutes a key intermediate for subsequent investigations within SEAFAIRER, including complementary analytical campaigns with additional EU feedstocks, blending studies, detailed analytics, and engine- or component-relevant performance testing relevant to marine fuel applications, as well as integration in existing refinery infrastructure (*e.g.*, co-processing). Three biogenic feedstocks were processed to generate representative batches: (i) biowaste sieving material, (ii) digestate derived from biowaste, and (iii) bagasse from *Agave tequilana* ("agave bagasse"). Supply was organised by SAGAVA and DTU, together with Fraunhofer UMSICHT. The resulting basic analytical data is summarised in chapter 4. As an illustrative example, the **bio-oil produced from biowaste sieving material in an experimental campaign yielded 7.9 wt% oil**. Its water content was measured at 4.5 wt% prior to centrifugation and decreased to 2.1 wt% after centrifugation, indicating effective removal of entrained water/aqueous phase. The corresponding **higher heating value** (HHV) was **36.83 MJ/kg**[1].

Overall, the experimental bio-oil batches were successfully produced and have been distributed to the SEAFAIRER project team for continued evaluation, with the main downstream testing activities planned and/or executed within WP6 (led by GALP) and WP7 (led by FVTR).

[1] Measurement taken: 36,826.3 J/g.

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3. Method for production and analysis of bio-oils

3.1 Description of trials on pilot unit

The experimental work reported in this Deliverable was conducted on the pilot unit at Fraunhofer UMSICHT's industrial facilities. For these trials (Task 3.1), the three specified feedstocks were first conditioned via drying and pelletisation to ensure consistent handling and stable operation during processing. A series of preliminary trials was performed in short, daily campaigns to establish suitable operating conditions and to define the process parameter window for each feedstock. Based on the outcomes of these scoping runs, **extended production campaigns of 2-4 days were subsequently carried out to generate the experimental bio-oil quantities** required for distribution to the project partners and for downstream SEAFAIRER Work Packages. An overview of the pilot plant is provided in Figure 1.

Figure 1. Pilot unit at Fraunhofer UMSICHT industrial facilities..

The SEAFAIRER project utilises VINTER technology, which stands for Vertical Intermediate Thermochemical Conversion developed by Fraunhofer. It converts various biogenic feedstocks into three valuable products: carbon-negative biochar, biocrude oil (intermediate biofuel), and syngas. Under SEAFAIRER, the technology will be scaled to demonstration capacity at TRL 7. A major advantage of this thermochemical process is its broad flexibility in input materials, allowing the use of globally abundant biogenic residues that do not compete with food and feed.

3.2 Description of analytics performed

The following analytical methods were used to determine the quality of the bio-oil produced:

- **Karl Fischer titration:** A method for determining water content using the reaction of iodine with water in the presence of sulphur dioxide and an alcohol. In the coulometric version, iodine is generated electrochemically;
- **CHNS elemental analysis:** Determines the carbon, hydrogen, nitrogen and sulphur content of a sample by high-temperature combustion. The resulting gases (CO_2, H_2O, N_2, SO_2) are separated and quantified via thermal conductivity;
- **Bomb calorimeter:** Used to determine the calorific value of a sample. The substance is combusted in a sealed oxygen-rich chamber. The heat released warms a surrounding water bath. The temperature increase allows precise calculation of the sample's energy content;
- **Total Acid Number (TAN) titration:** Determines the amount of acidic compounds in oil samples through potentiometric titration with potassium hydroxide. The volume of titrant needed to reach the titration endpoint reflects the acid content.

For Karl Fischer titration, CHNS elemental analysis, bomb calorimetry, and TAN titration, the results given were calculated as the arithmetic mean of three independent parallel determinations.

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3.3 Pictures of bio-oils per feedstock

Figure 2 shows representative oil samples from the three feedstocks; biowaste sieving material, digestate from biowaste and agave bagasse.

Figure 2. From left to right: Bio-oil produced from biowaste sieving material, digestate derived from biowaste, and agave bagasse.

3.4 Description of sampling procedures

As it leaves the reactor, the process stream, consisting of gas, process water, and bio-oil, is diverted at a sampling point. Within the plant, the stream is cooled in a water-based heat exchanger, causing the condensable components to liquify, while the gas remains in the gaseous phase and exits the sampling line. The resulting condensate is collected and subsequently separated in a separatory funnel. The oil phase is then withdrawn for analysis. For experiments requiring larger quantities of oil, phase separation was carried out by means of conductivity control in a purpose-built separation station.

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4. Analytic report

The following bio-oil volumes were produced for distribution and subsequent testing within the SEAFAIRER project:

- Biowaste sieving material: ca. 627 litres (l);
- Digestate from biowaste: ca. 155 l;
- Agave bagasse: ca. 168 l

These batches were produced to support follow-up activities such as blending trials, complementary analytical work, and performance testing in the relevant Work Packages.

4.1 Biowaste sieving material

Table 1 summarises the results of the **Karl Fischer titration** conducted to quantify the water content of the bio-oil derived from biowaste sieving material. The **first row** reports the water content of the **centrifuged oil** (*i.e.*, after phase separation), representing the most relevant value for downstream handling and fuel-related testing. Subsequent entries reflect measurements from earlier processing stages (where applicable) for comparison.

Table 1. Measurement results of Karl Fischer titration.

Titration H_2O			
Measurement 1	Measurement 2	Measurement 3	MV [%]
1.73	1.8	1.72	1.75

Table 2 summarises the average elemental composition of the feedstock, produced bio-oil, and resulting biochar, determined by CHNS elemental analysis. Reported values are given in wt%. Oxygen (O) is provided as the remaining fraction (*i.e.*, calculated by difference to 100 wt%, where applicable). All entries marked with an asterisk (*) represent average values calculated from the underlying measurements

Table 2. Measurement results of CHNS elemental analysis.

CHNS elemental analysis					
Sample	N [%]	C [%]	H [%]	S [%]	O [%]
Feed*	1.85	39.87	15.14	0.15	53.09
Oil*	4.33	78.04	9.02	0.34	8.27
Char*	1.49	42.12	0.93	0.15	55.31

Table 3 below reports the average bomb calorimetry results for feedstock, bio-oil, and char. For clarity and consistency with common fuel-property reporting, the calorific values have been converted from J/g to MJ/kg. All entries marked with an asterisk (*) represent average values calculated from the underlying measurements.

Table 3. Average HHV determined by bomb calorimetry.

Bomb calorimetry	
Sample	Calorific value [MJ/kg]
Feed*	16.214
Oil*	36.743
Char*	15.401

Table 4 presents the results of the total acid number (TAN) determination for the bio-oil after centrifugation, measured by titration. Three replicate measurements were performed to assess repeatability, and the mean value (MV) is reported as the average of the three results.

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Table 4. Total acid number (TAN) of centrifuged bio-oil determined by titration.

Titration TAN			
Measurement 1	Measurement 2	Measurement 3	MV
14.66	9.86	9.16	11.23

4.2 Digestate derived from biowaste

Table 5 summarises the results of the **Karl Fischer titration** conducted to quantify the water content of the bio-oil derived from digestate. The **first row** reports the water content of the **centrifuged oil** (*i.e.*, after phase separation), representing the most relevant value for downstream handling and fuel-related testing. Subsequent entries reflect measurements from earlier processing stages (where applicable) for comparison.

Table 5. Measurement results of Karl Fischer titration.

Titration H_2O			
Measurement 1	Measurement 2	Measurement 3	MV [%]
2.17	2.5	1.97	2.22

Table 6 summarises the average elemental composition of the feedstock, produced bio-oil, and resulting char, determined by CHNS elemental analysis. Reported values are given in wt%. Oxygen (O) is provided as the remaining fraction (i.e., calculated by difference to 100 wt%, where applicable). All entries marked with an asterisk (*) represent average values calculated from the underlying measurements

Table 6. Measurement results of CHNS elemental analysis.

CHNS elemental analysis					
Sample	N [%]	C [%]	H [%]	S [%]	O [%]
Feed*	2.67	30.8	3.98	0.73	61.83
Oil*	4.12	77.78	8.99	0.60	8.5
Char*	1.16	47.6	0.91	0.29	50.04

Table 7 below reports the average bomb calorimetry results for feedstock, bio-oil, and char. For clarity and consistency with common fuel property reporting, the calorific values have been converted from J/g to MJ/kg. All entries marked with an asterisk (*) represent average values calculated from the underlying measurements.

Table 7. Average HHV determined by bomb calorimetry.

Bomb calorimetry	
Sample	Calorific value [MJ/kg]
Feed*	12.239
Oil*	36.333
Char*	15.370

Table 8 presents the results of the total acid number (TAN) determination for the bio-oil after centrifugation, measured by titration. Three replicate measurements were performed to assess repeatability, and the mean value (MV) is reported as the average of the three results.

Table 8. Total acid number (TAN) of centrifuged bio-oil determined by titration.

Titration TAN			
Measurement 1	Measurement 2	Measurement 3	MV
5.75	5.68	5.64	5.69

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4.3 Agave bagasse

Table 9 summarises the results of the **Karl Fischer titration** conducted to quantify the water content of the bio-oil derived from agave bagasse. The **first row** reports the water content of the **centrifuged oil** (*i.e.*, after phase separation), representing the most relevant value for downstream handling and fuel-related testing. Subsequent entries reflect measurements from earlier processing stages (where applicable) for comparison.

Table 9. Measurement results of Karl Fischer titration.

Titration H_2O			
Measurement 1	Measurement 2	Measurement 3	MV [%]
8.19	7.82	8.14	8.05
1.47	1.52	1.54	1.51

Table 10 summarises the average elemental composition of the feedstock, produced bio-oil, and resulting char, determined by CHNS elemental analysis. Reported values are given in wt%. Oxygen (O) is provided as the remaining fraction (i.e., calculated by difference to 100 wt%, where applicable). All entries marked with an asterisk (*) represent average values calculated from the underlying measurements

Table 10. Measurement results of CHNS elemental analysis.

CHNS elemental analysis					
Sample	N [%]	C [%]	H [%]	S [%]	O [%]
Feed*	0.54	41.67	5.64	0	52.15
Oil*	1.72	80.06	9.16	0.16	8.9
Char*	1.00	61.87	1.63	0.01	35.49

Table 11 below reports the average bomb calorimetry results for feedstock, bio-oil, and char. For clarity and consistency with common fuel property reporting, the calorific values have been converted from J/g to MJ/kg. All entries marked with an asterisk (*) represent average values calculated from the underlying measurements.

Table 11. Average HHV determined by bomb calorimetry.

Bomb calorimetry	
Sample	Calorific value [MJ/kg]
Feed*	16.543
Oil*	36.305
Char*	21.849

Table 12 presents the results of the total acid number (TAN) determination for the bio-oil after centrifugation, measured by titration. Three replicate measurements were performed to assess repeatability, and the mean value (MV) is reported as the average of the three results.

Table 12. Total acid number (TAN) of centrifuged bio-oil determined by titration.

Titration TAN			
Measurement 1	Measurement 2	Measurement 3	MV
1.73	1.68	No measurement	1.71

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5. Conclusion

This Deliverable confirms the **successful production of the experimental bio-oil batches in the pilot unit from (i) biowaste sieving material, (ii) biowaste-derived digestate, and (iii) agave bagasse**, and documents their initial quality through a consistent set of screening analyses (water by Karl Fischer, CHNS elemental composition, calorific value by bomb calorimetry, and acidity via TAN titration). These measurements provide the first, comparable dataset needed to (a) validate batch-to-batch consistency, (b) support logistics, and (c) enable downstream partner activities (blending, advanced analytics, and marine and fuel co-processing-oriented testing).

Across the produced bio-oils, the applied analytical suite captures several parameters that are particularly relevant for marine fuel development pathways. **Water content and acidity are widely recognised as key "gatekeeper" properties for thermochemical bio-oils because they influence phase behaviour, corrosion risk, compatibility in blends, and storage stability**. The centrifugation step demonstrably reduced the water content in the illustrative case (biowaste sieving material), indicating that a simple post-treatment can already improve the oil's suitability as well as handling and testing. Likewise, TAN quantifies the total acidic functionality in the oil (reported as mgKOH/g), which is commonly used to track acidity-driven risks and changes during storage and processing; this metric is routinely applied for bio-oils using established titration methodologies.

The **CHNS and calorific value results further support the role of these batches as meaningful test materials for SEAFAIRER.** The comparatively high energy content measured for the bio-oil (relative to its corresponding feed and char) indicates a concentrated energy carrier suitable for evaluating blending strategies and combustion-related behaviour, while the elemental composition highlights the fundamental compositional shift from biomass to bio-oil and biochar. At the same time, **these results also support the strong advantages of the SEAFAIRER bio-oil produced against other thermochemical processes** (*e.g.*, raw pyrolysis, HtL, *etc.*), which typically remain chemically complex and reactive due to high oxygenated compound content; without further quality management (*e.g.*, stabilisation/upgrading/hydrotreatment), properties such as acidity, water content, and storage instability can directly limit use as marine fuel.

In the **marine context, ISO 8217 provides the overarching framework for marine fuel specifications** (including renewable and bio-derived components) and is specifically defined for fuels supplied prior to onboard treatment steps such as centrifugation and filtration. **The successful generation and distribution of these SEAFAIRER bio-oil batches therefore represents an important enabling step**: it provides project partners with representative materials to assess blending and move these oils toward practical marine application, consistent with the evolving ISO 8217:2024[2] landscape, which will be further investigated in Work Package 7.

[2] International Organization for Standardization. (2024). ISO 8217:2024 Products from petroleum, synthetic and renewable sources—Fuels (class F)—Specifications of marine fuels (7th ed.). ISO.

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