# Pulp to fuel via HTL and IHTL

*BL2F Final Event*
*6.3.2024*

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BL2F
Transforming Black Liquor to Biofuel

This project has received funding from the European Union Grant Number 884111

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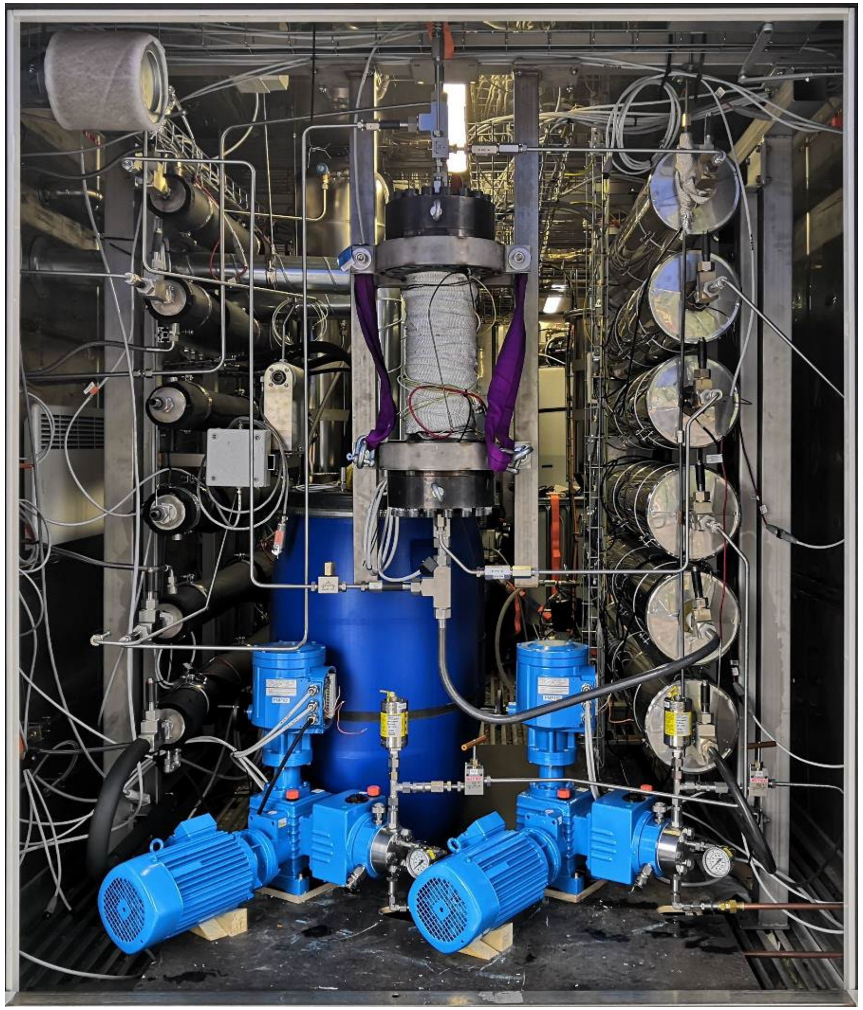

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# Hydrothermal liquefaction

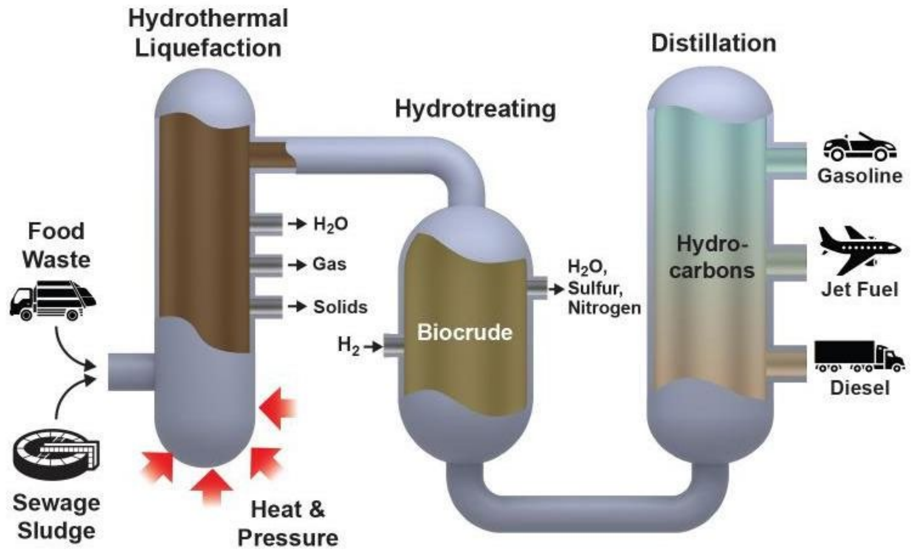


Illustration by Michael Perkins | Pacific Northwest National Laboratory

**Benefits**

- High quality and high yield of biocrude
- Different feedstock
- Drying of wet biomass is not required

**Challenges**

- High temperature and pressure requires extra opex and capex
- Pre-treatment of different feedstock
- Flexible reactor and process design for varying feedstocks

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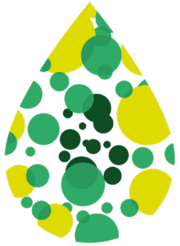
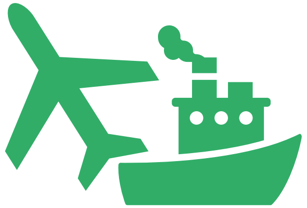
# BL2F Project Goals



**Create a high-quality drop-in biofuel**



**Decrease carbon emissions from aviation and shipping**

**Decrease the use of fossil fuels**

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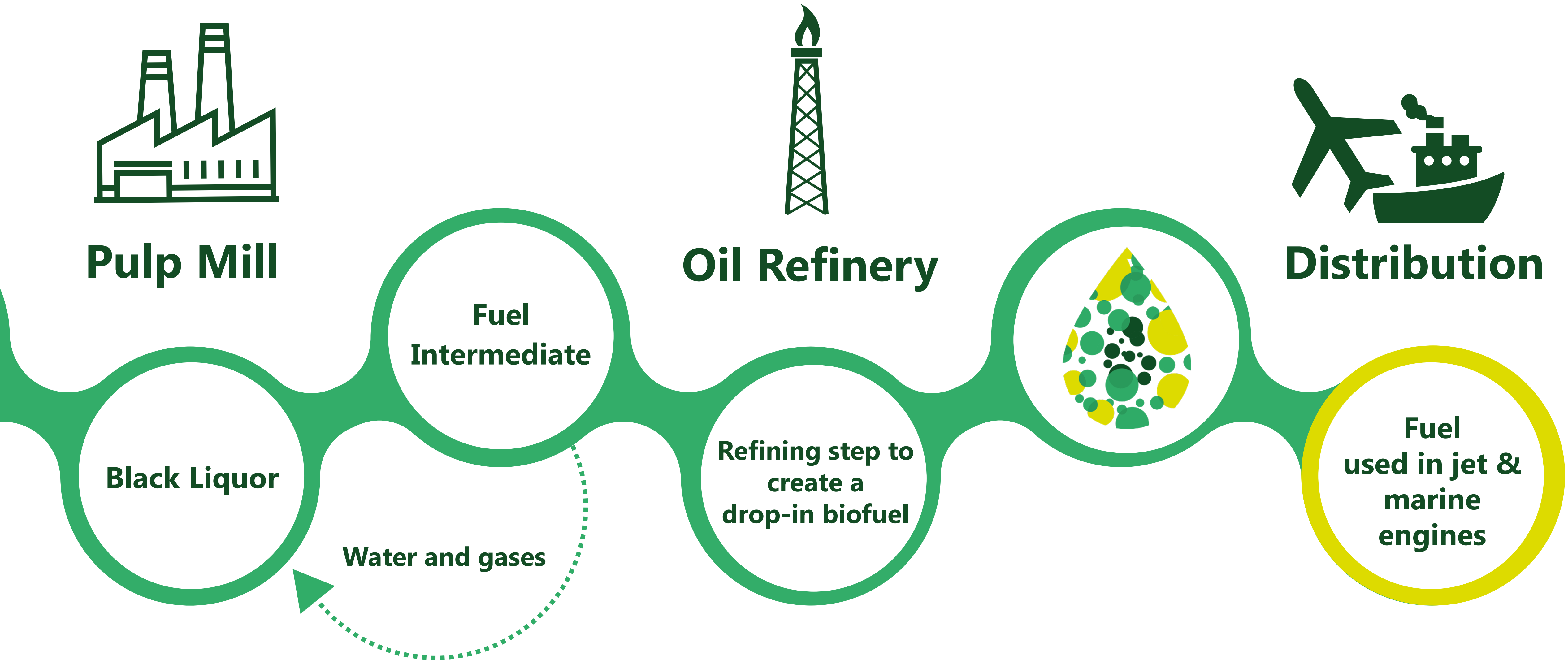
# The BL2F Process

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# There is still a long way to go...

BL2F is here

**2020 – 2023**

**TRL-3** Experimental proof of concept

**TRL-4** Technology validated in lab

**TRL-5** Technology validated in relevant environment

**2023 – 2025**

**TRL-6** Technology demonstrated in relevant environment

**TRL-7** System prototype

**2026 – 2027**

**TRL-8** System complete and qualified

**FIRST COMMERCIAL PLANT BUILT**

**2028**

**TESTING**

**2029+**

**TRL-9**

**BL2F SOLUTION ON THE MARKET**

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Karlsruhe Institute of Technology

Tampereen yliopisto
Tampere University

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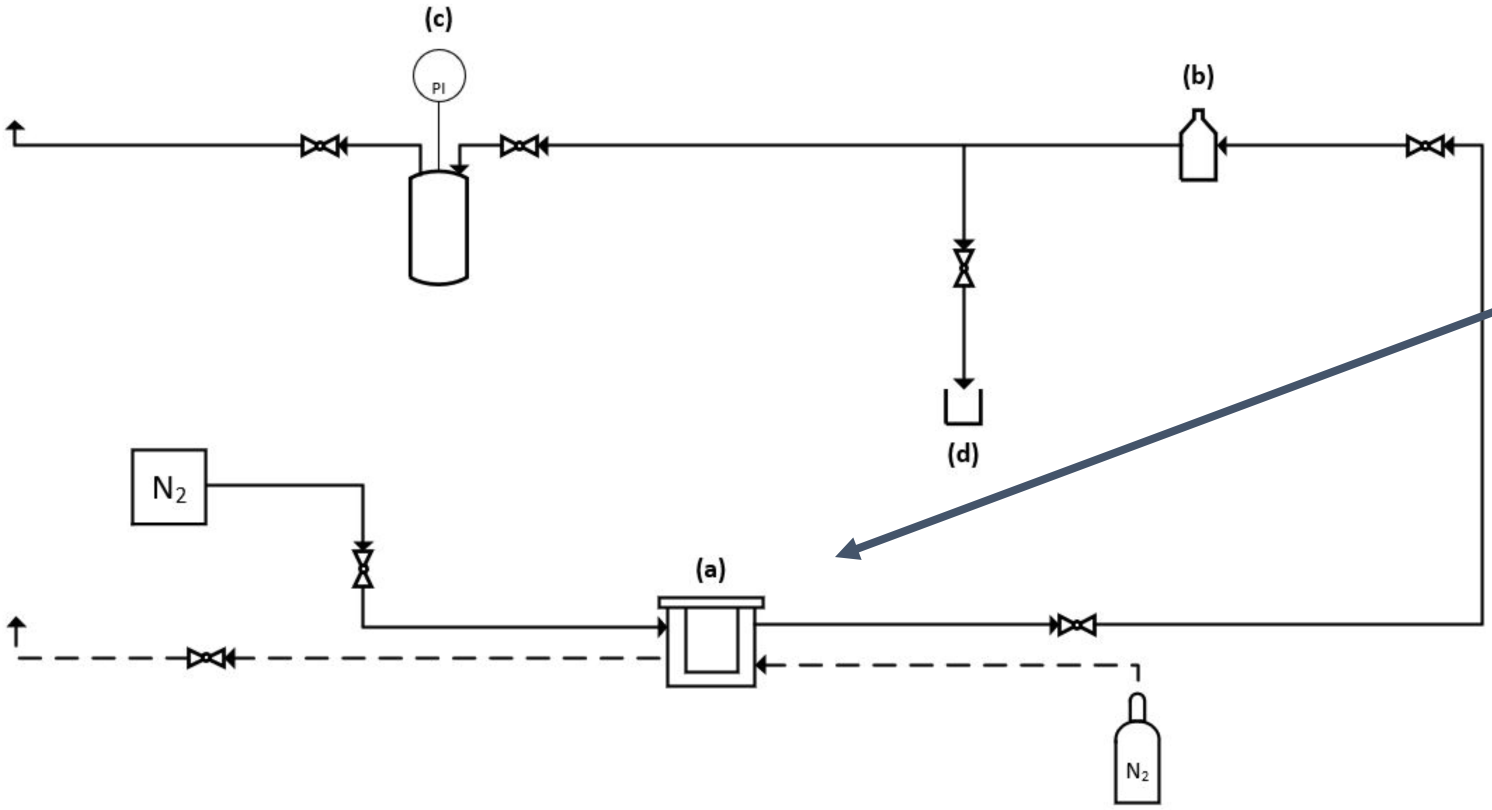
# Laboratory-scale experiments

# Experimental Setup (Batch)




Micro autoclaves, $V = 25$ mL

Heating process in sand bath

- $t_H = 10$ min

Investigated conditions:

- $T_R = 250 – 400$ °C ($t_R = 20$ min)
- $t_R = 0 – 30$ min ($T_R = 325$ °C, 375 °C)
- Pressure: around 200 – 250 bar (via fill level)
- $HS^-$ concentration: 0 g/L, 1 g/L, 2 g/L, 3 g/L

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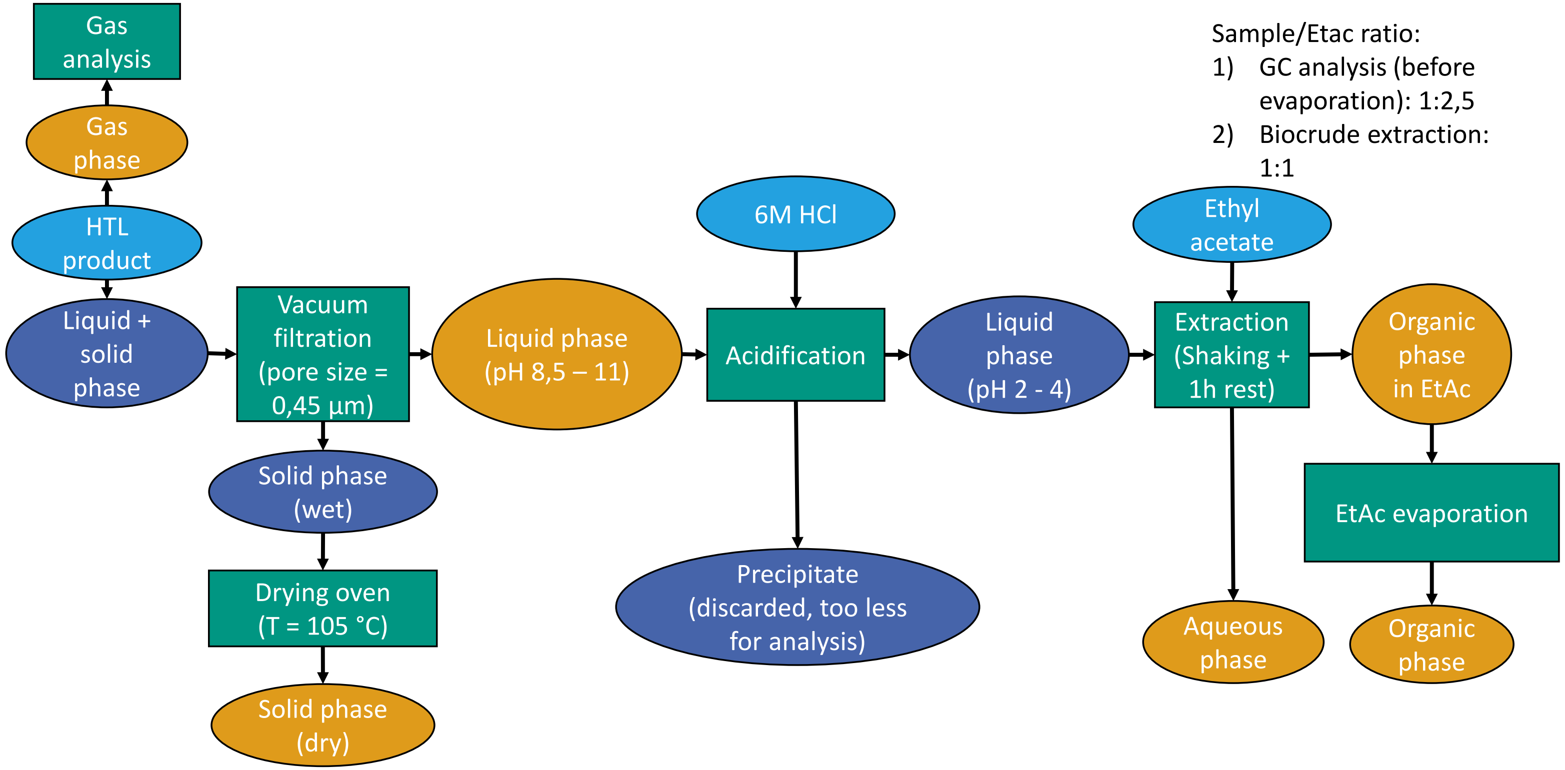
# Extraction procedure

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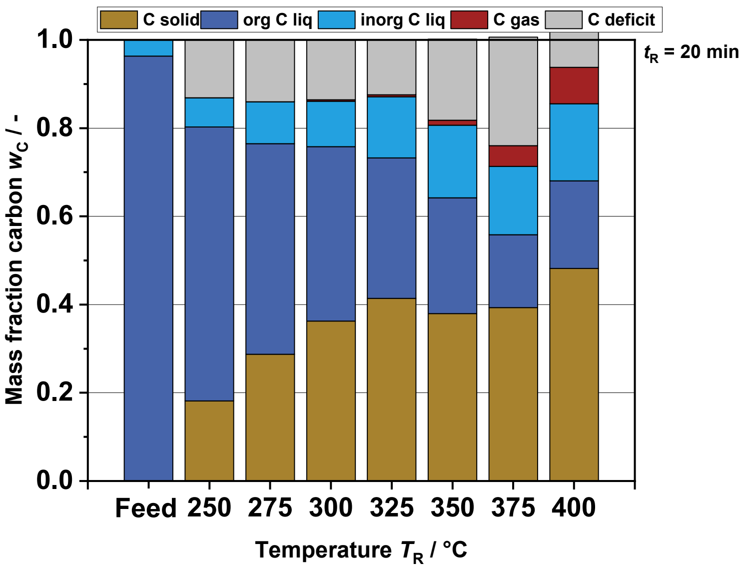
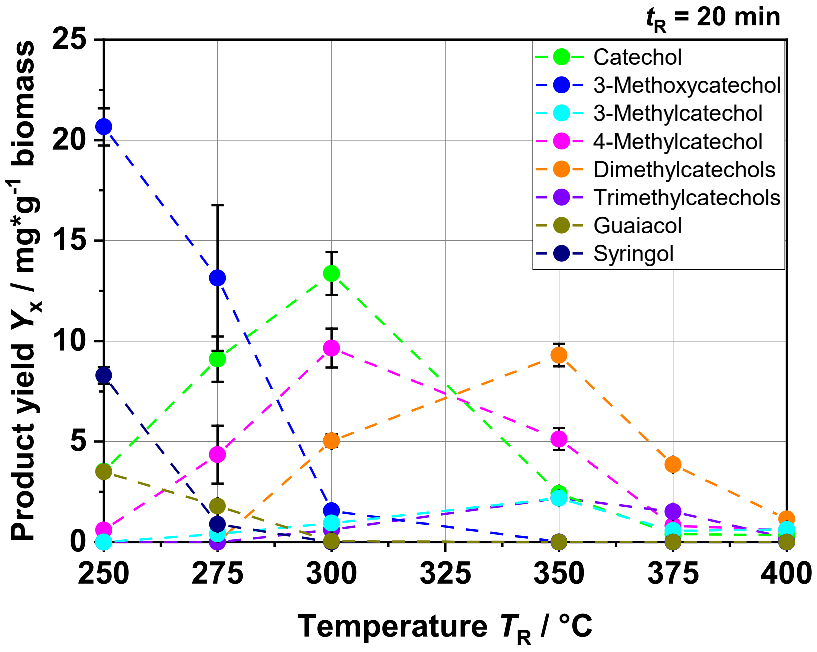
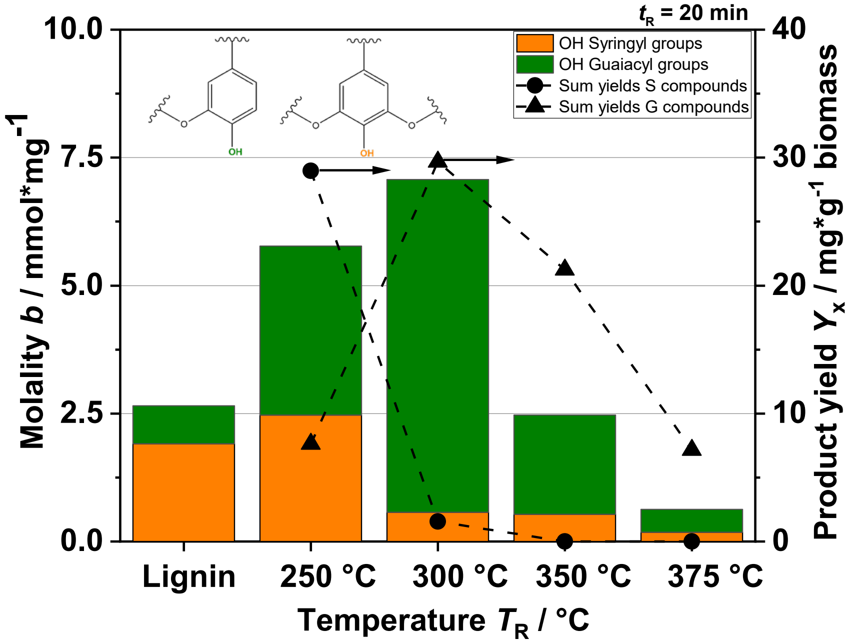
# Results of Batch experiments




- Organic carbon in liquid phase decreasing with $T_R$ ↑
- High carbon yield in solid fraction

- Main aromatic monomer compounds: catechols
- Reaction pathway:
  $-OCH_3$ -> -OH -> -OH + $-CH_3$

- $^{31}P$ NMR results of biocrude fit well with GC-FID results
- Monomer and oligomer reactions at functional groups are similar

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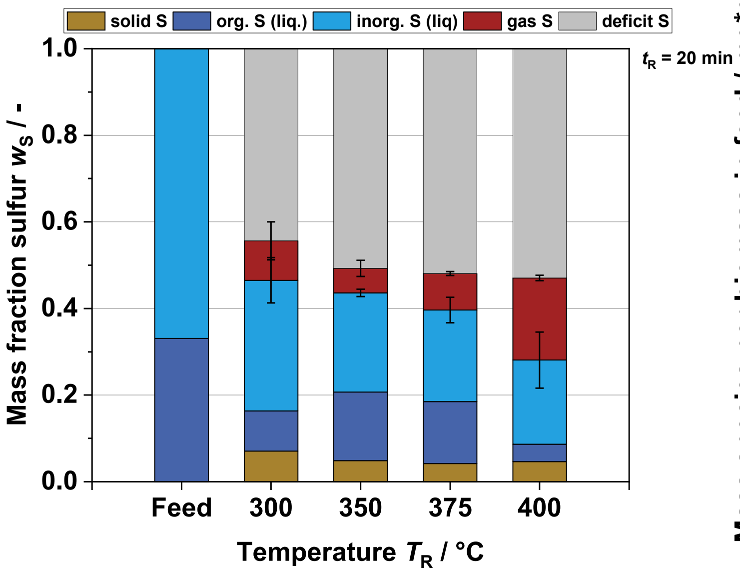
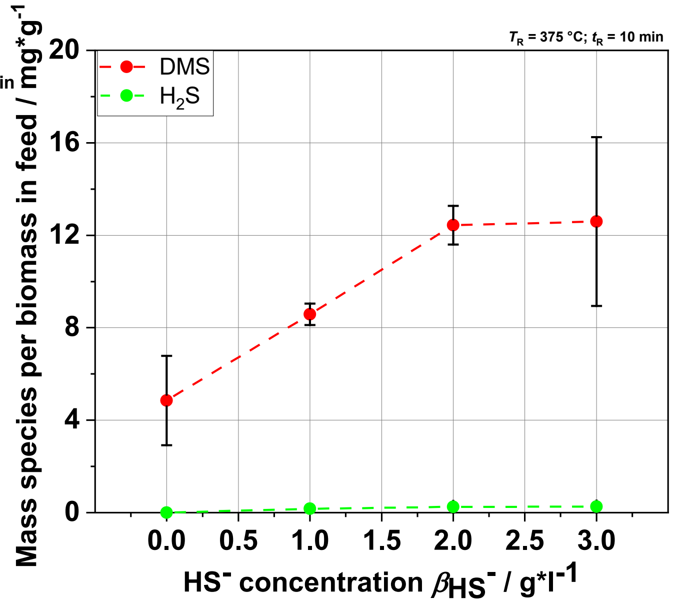
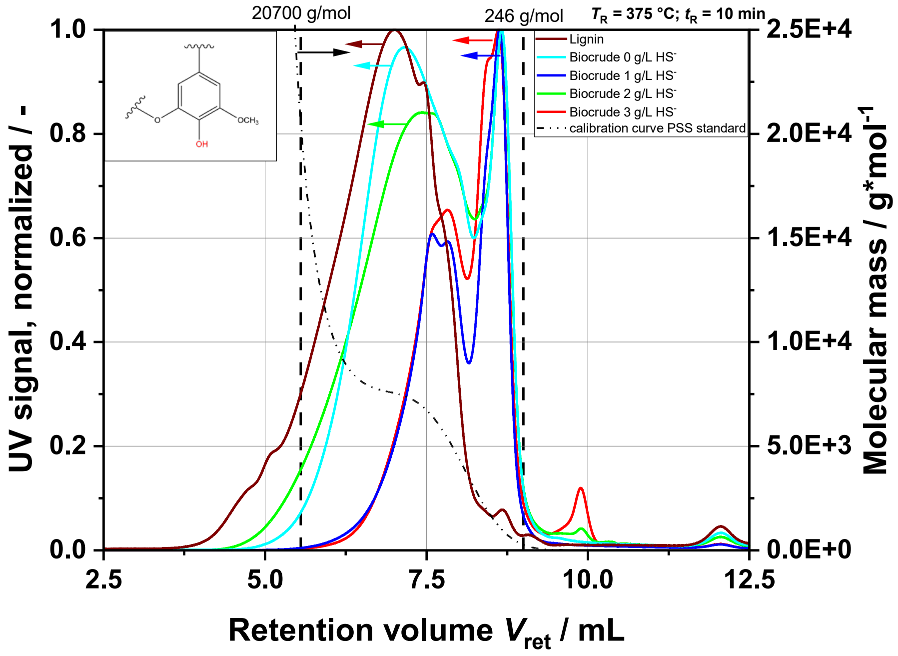
*These results will be published in ACS Energy & Fuels: Wörner et al. From Pulp to Aromatic Products Reaction Pathways of Lignin Depolymerization; DOI: 10.1021/acs.energyfuels.3c04509*

# Influence of sulfide ($HS^-$)




- Inorganic sulfur reduces drastically compared to S in feed
- Reactions between inorganic sulfur salts and organic matter

- Increasing $HS^-$ concentration in feed leads to higher organo-sulfur yields
- Main organo-S-compound: dimethylsulfide (DMS)

- Increasing $HS^-$ concentration in feed accelerates depolyermerization of lignin

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*These results will be published in ACS Energy & Fuels: Wörner et al. The Impact of Sulfur-containing Inorganic Compounds during the Depolymerization of Lignin by Hydrothermal Liquefaction of Black Liquor; DOI: 10.1021/acs.energyfuels.3c04737*

# Lab tests summary

- Catechols main aromatic products in biocrude
- Yields for aromatic compounds and biocrude are decreasing with $T_R\uparrow$
- Influence of salts (here $HS^-$) was observed, salt separation could minimize it
- Extraction procedure of biocrude is an important topic
  - Further studies showed that a lot of organics are adsorbed on the solid phase
  - An extraction from solid and liquid phase together could lead to higher yields

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# Continuous HTL facility developed at Tampere University

# Continuous Reactor Facility - EHTA

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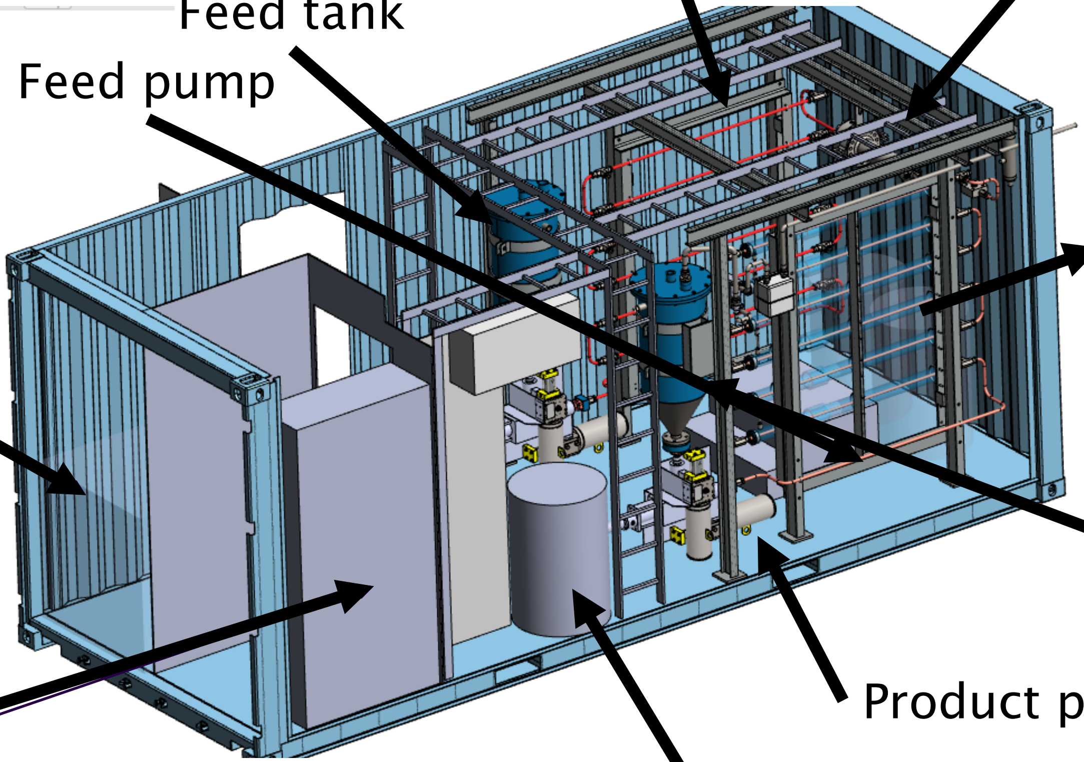
| Process conditions | Maximum value |
|---|---|
| Temperature | 400 °C |
| Pressure | 300 bar |
| Feeding rate | 13.2 l/h |



# BL2F Reactor

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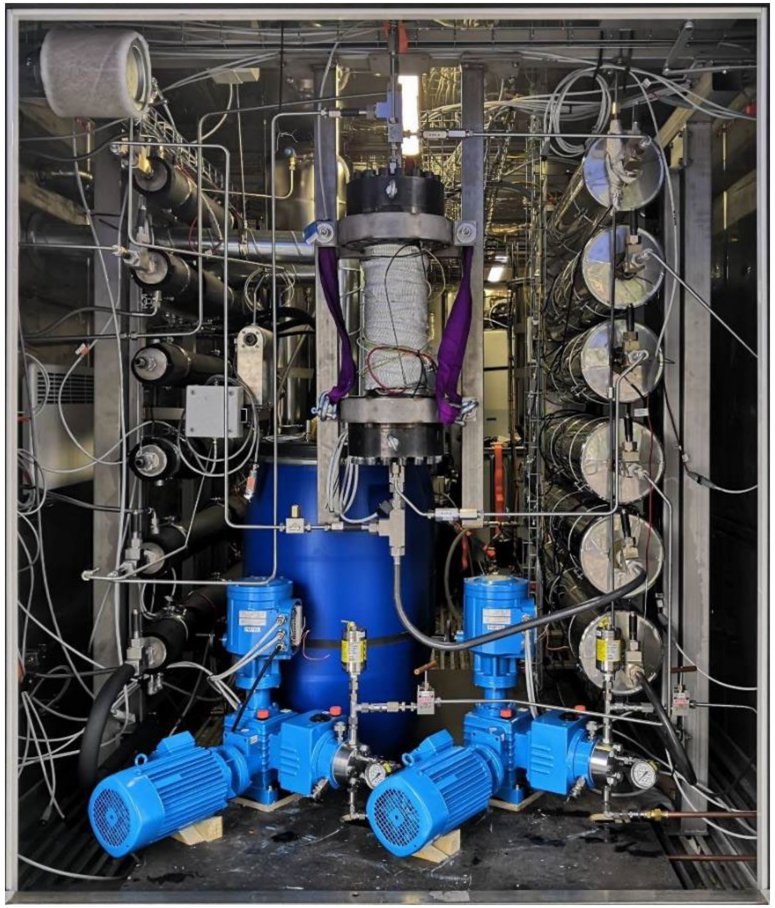

# First result from our Continuous experiments

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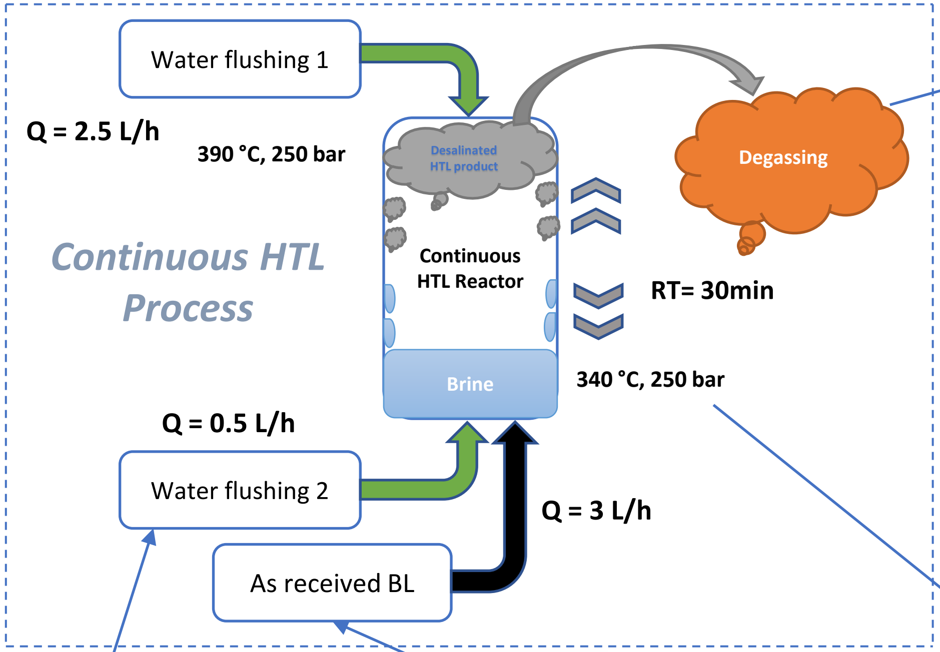



Coolers for Top product

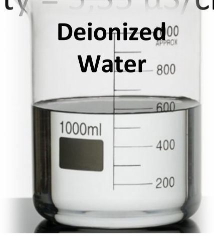
Q = 1.8 L/h


Top Product

Due to the gravity forces Most of the produced Hydrochar is transferred to the bottom product (Brine).

**Deionized Water :**
pH = 6,52 ;

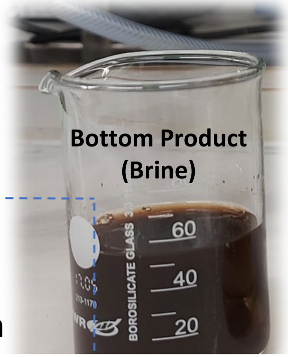
Conductivity = 5,35 µS/cm





**Black Liquor:** pH = 13,4 ;

Conductivity = 68,9 mS/cm




Coolers for Bottom product (Brine)

Q = 4 L/h

*pH and Conductivity of Brine & HTL Top Product*

**HTL Product:** pH = 8,89 ; Conductivity = 15,17 mS/cm

**Brine:** pH = 9,37 ; Conductivity = 41,8 mS/cm

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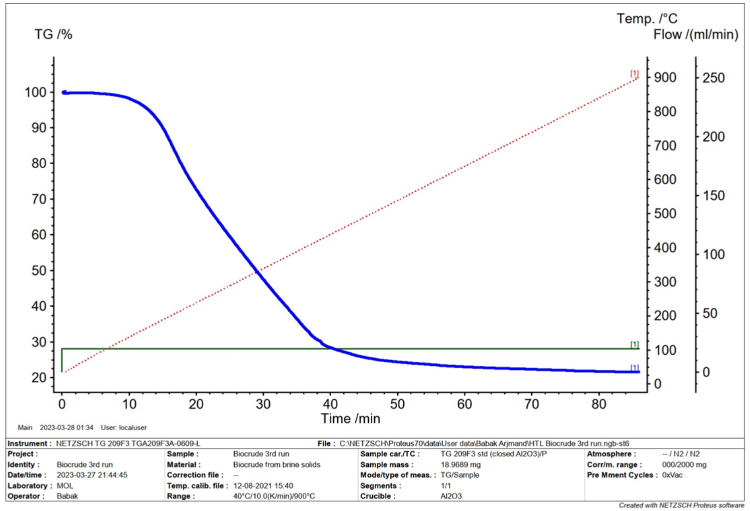
# Elemental Analysis (Biocrude)

| C | H | N | S | O | Ash |
|---|---|---|---|---|---|
| 79.337 | 7.29 | 0.025 | 2.214 | 10.76 | 0.37 |

**The disappearance of alcohol and ether moieties was in accordance with low oxygen content obtained from elemental analysis.**

# Thermogravimethric Analysis (Biocrude)




| Moisture | VM | FC | Ash |
|---|---|---|---|
| 2,7 | 77.3 | 19.63 | 0.37 |

# Conclusion

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- HTL: liquefaction of carbon and hydrogen containing feedstocks
  - Advantage: very moist and low-grade feedstocks can be used
- BL2F project: Basics of design for larger-scale HTL demontration process
  - Black liquor as fuel, integration with chemical pulp mill
  - Biocrude oil to drop-in fuels (shipping, aviation)
  - Pilot plant testing (EHTA plant) at Tampere Unversity campus
- Pilot testing:
  - Mechanical challenges have caused schedule delays
  - Biocrure oil samples generated for further testing and processing
- Future: Long way to go to commercial (Aimed market-readiness 2029)

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# BL2F Partners:

Tampere University

Brunel University London

KIT Karlsruhe Institute of Technology

SINTEF

PAUL SCHERRER INSTITUT PSI

VTT

THE NAVIGATOR COMPANY

Valmet

NESTE

Ranido

LGi sustainable innovation

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# Thank you!

# Get in touch with the project:

- Coordinator: Prof. Dr. Tero Joronen, Tampere University
- Website: www.bl2f.eu

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