Norwegian Centre for Environmentfriendly Energy Research

Bio4Fuels

Norwegian Centre for Sustainable Bio-Based Fuel and Energy

Continuous vs. Batch hydrothermal liquefaction of Nordic biomass

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Overview

Introduction And Background

Our equipment

Continuous vs Batch

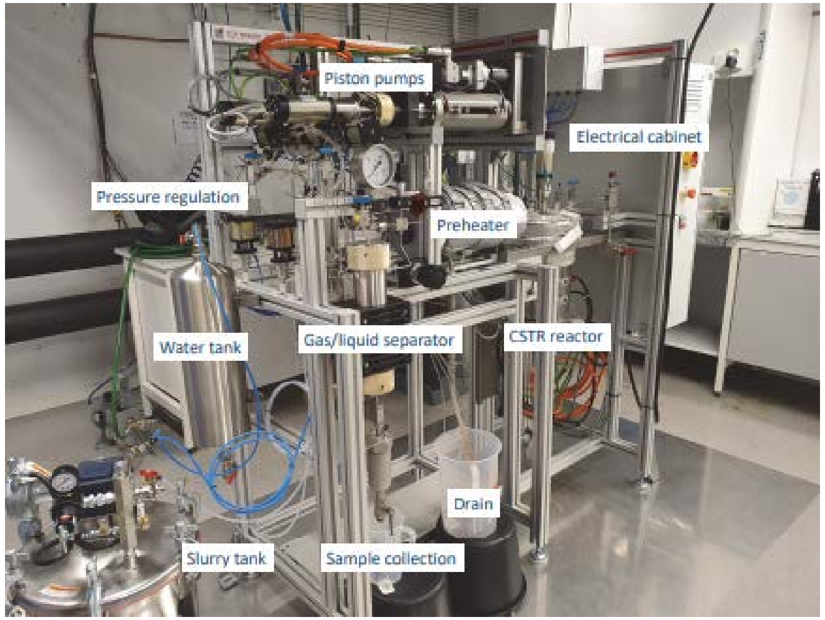
Research Roadmap (WP2.2 Hydrothermal Liquefaction)

Results

Hurdles and Final Steps

Why do we need oil from biological materials?

- Renewable drop-in fuels are more relevant for fossil fuel replacement in many sectors
- Use of biomass residues is not only responsible but necessary for a circular economy
- HTL relies on the same techniques as fossil fuels with shorter times and better inputs



Refresher on Hydrothermal Liquefaction

- Hydrothermal processes: Supercritical or subcritical conditions
  - Increases solubility of organics
  - Water acts as solvent, reactant, and catalyst

Critical point of water:
374 °C, 220 bar

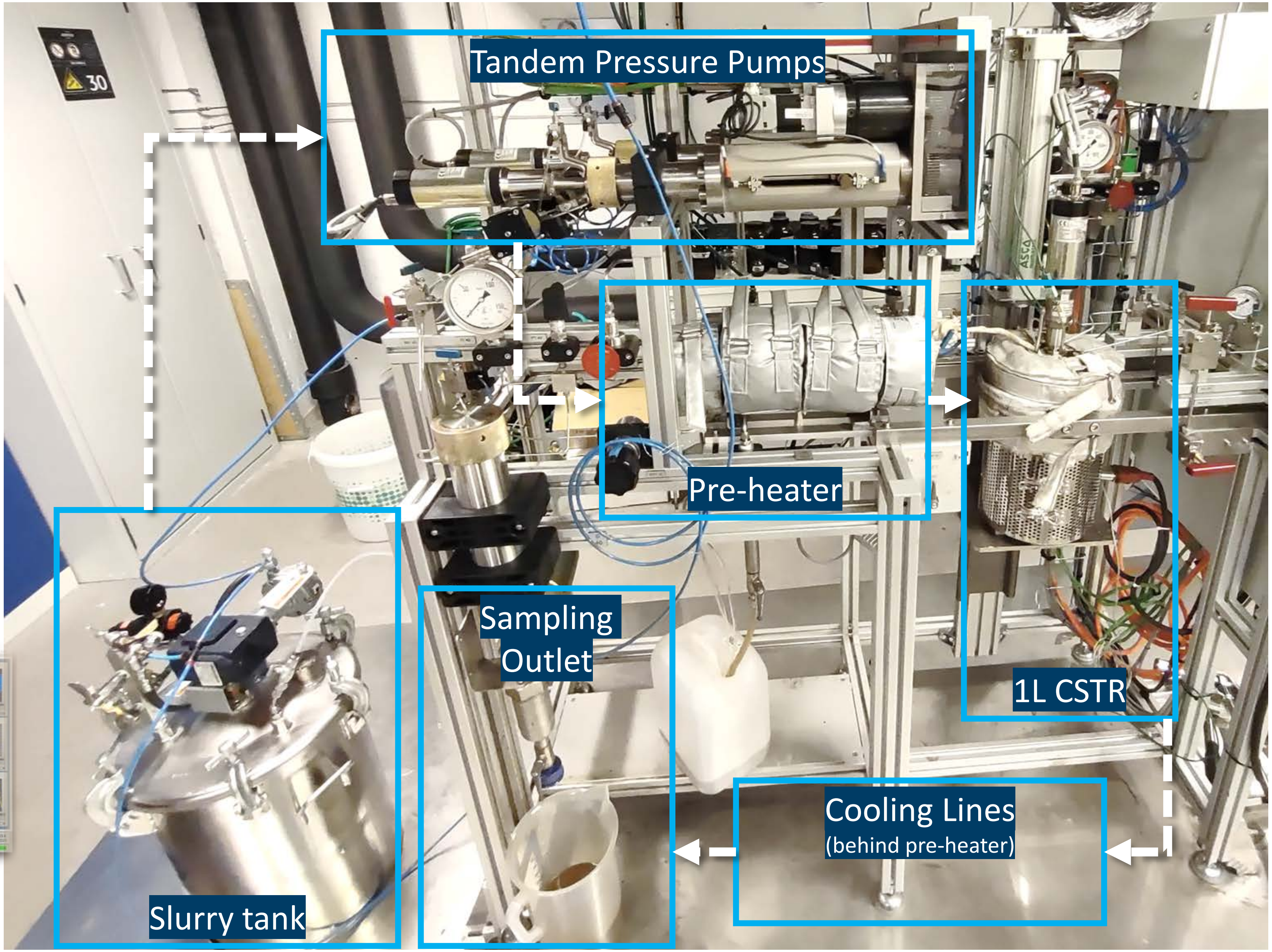
Wood powder slurry

Our research:
350 °C, 300 bar
400 °C, 300 bar

Raw bio-oil product

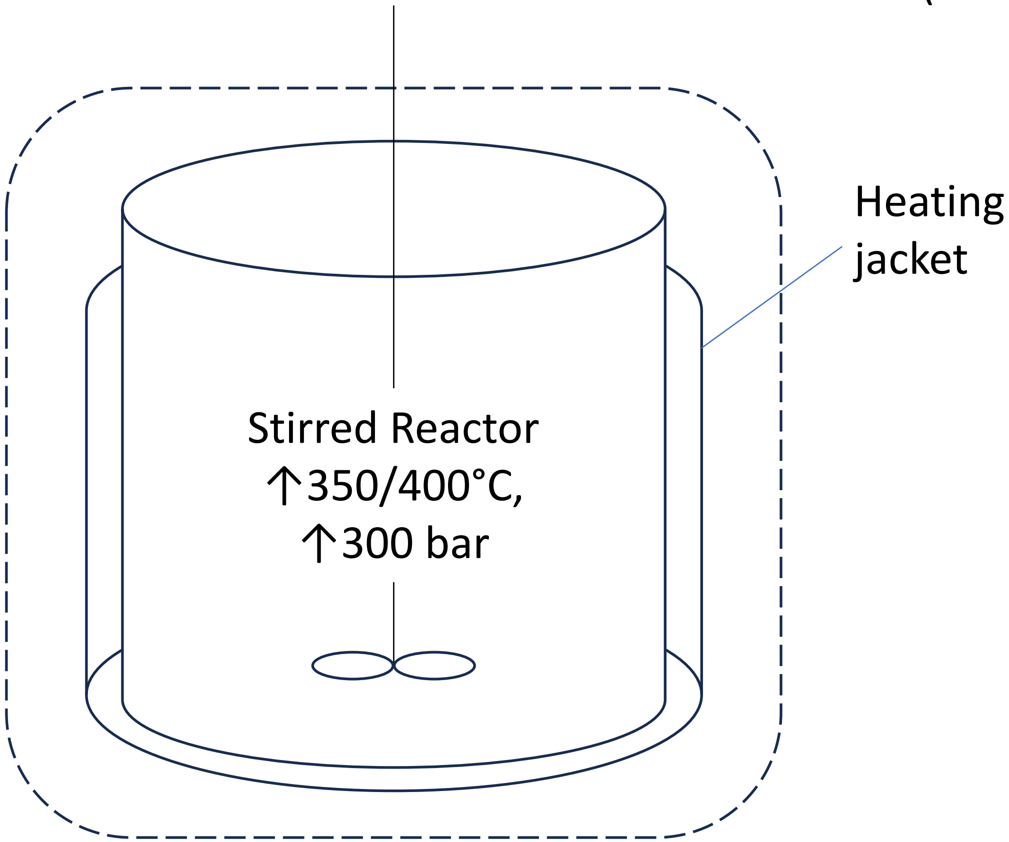
Speed and pressure created by Pressure pumps

All systems tracked on connected PC

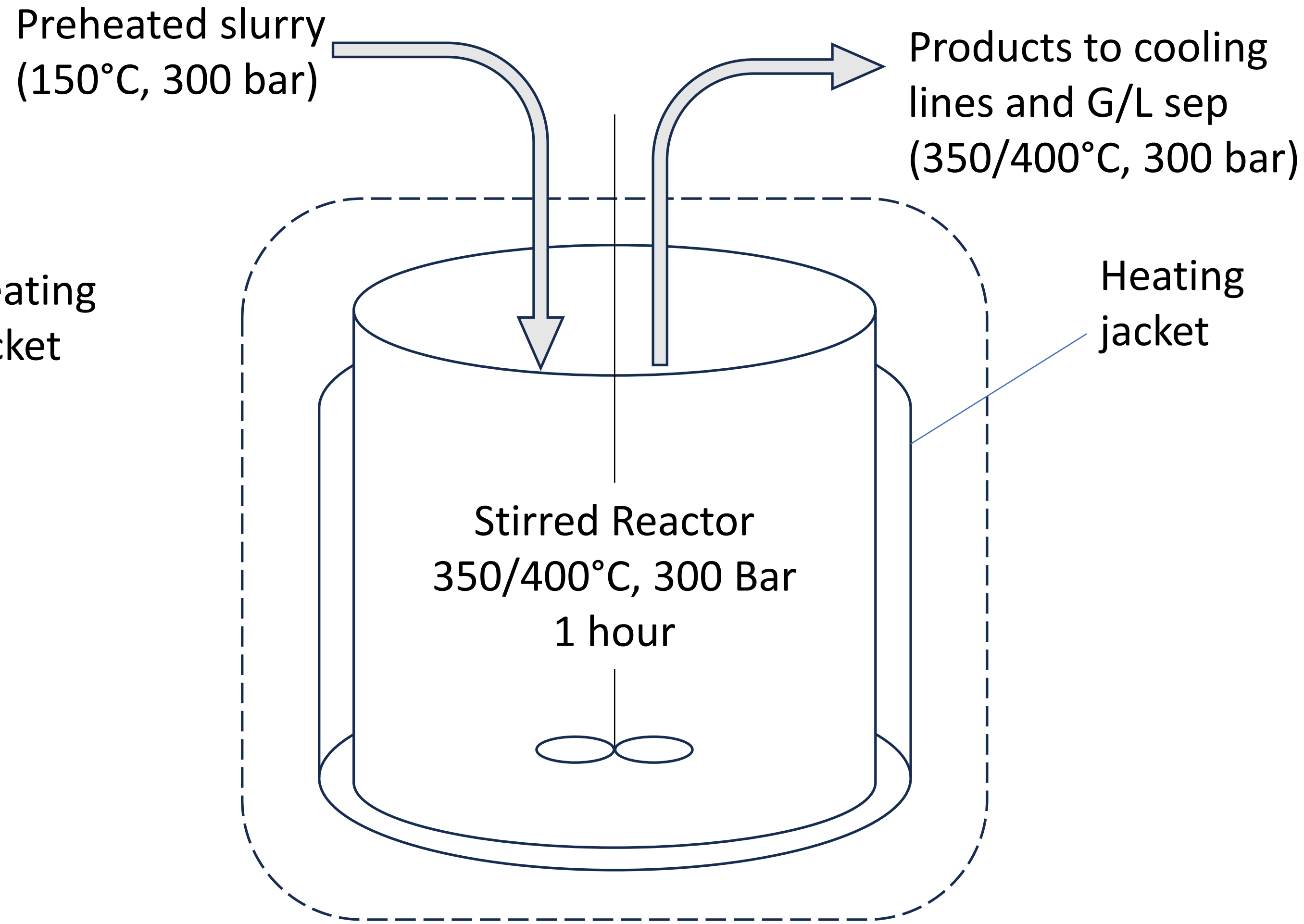


Continuous Reactor for Hydrothermal Experiments

Continuous vs Batch



Purged with Nitrogen
Sealed and heated
Cooled before products removed



Steady state
Temperature & Pressure constant
Residence time adjustable

Separation of products

Product mix from reactor

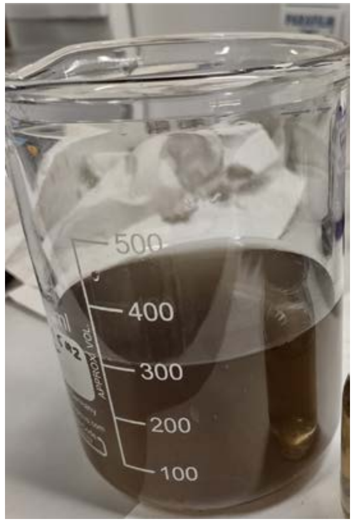
1 hour residence time

1. Initial separation of water and oil
2. Filtering of hydrochar in reactor
3. Extraction of organics/oil from water phase

Separated products

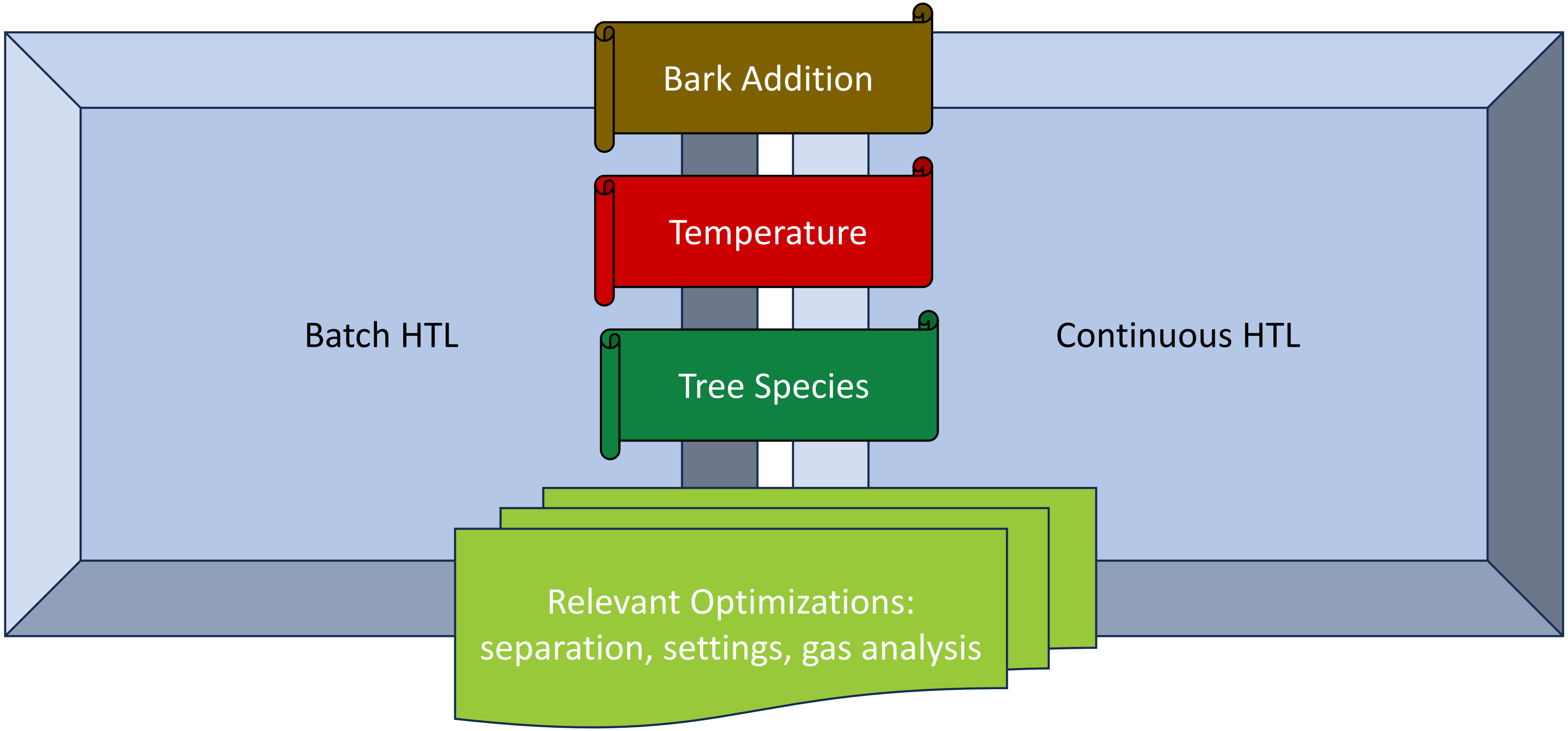
Raw bio-oil

Hydrochar



Aqueous

Research Roadmap



Batch HTL Research for Bio4Fuels

- Investigate effects of...
  - <u>Temperature</u> (350 °C vs. 400 °C)
  - <u>Feedstock</u> (gran vs. furu)
  - <u>Inclusion of bark</u> (5 wt.%, 10 wt.%, 15 wt.%)
  - Separation tests with toluene

Slurry ingredients: wood powder, bark, water, catalyst

Slurry to batch reactions

Slurry to continuous reactions

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Batch Results

Hydrothermal Liquefaction of Bark-containing Nordic Biomass

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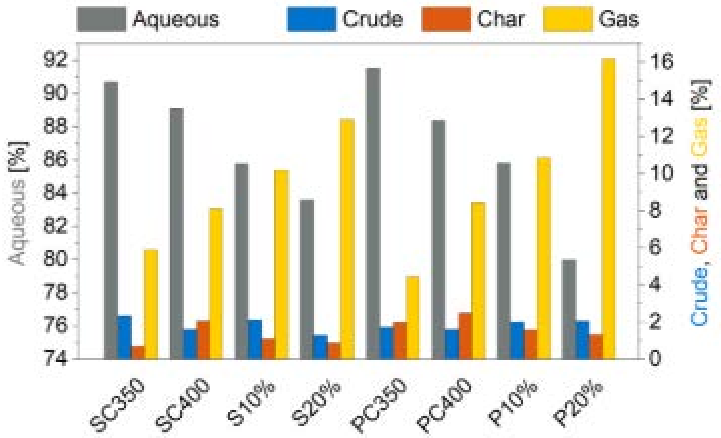
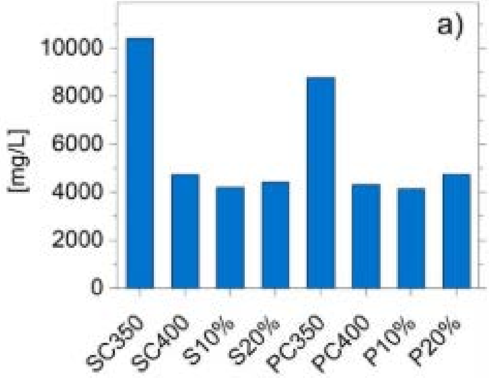
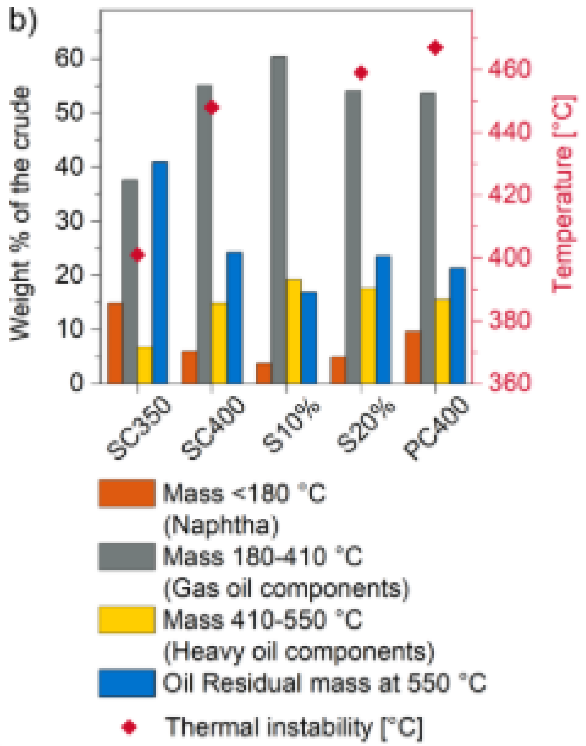
- Higher yields from Pine than from Spruce
- Inclusion of bark does not result in noticeable changes in product yields
- Higher temperatures resulted in more C-O groups and lower temps had higher aromatics
- Much of the weight of biocrudes is from a range of gas oil components
- Oil from supercritical conditions (400C) was of higher quality than subcritical (350C)
- Organics in aqueous phase depended primarily on temperature







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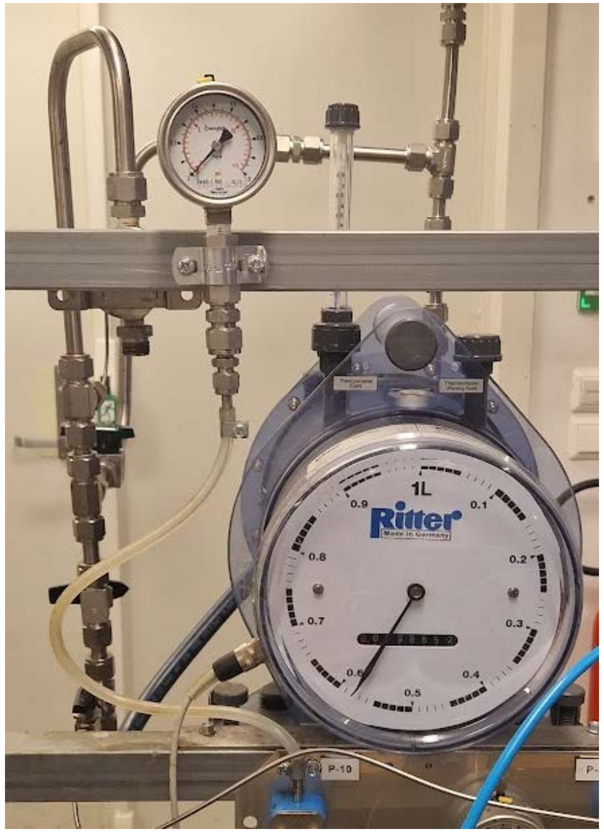
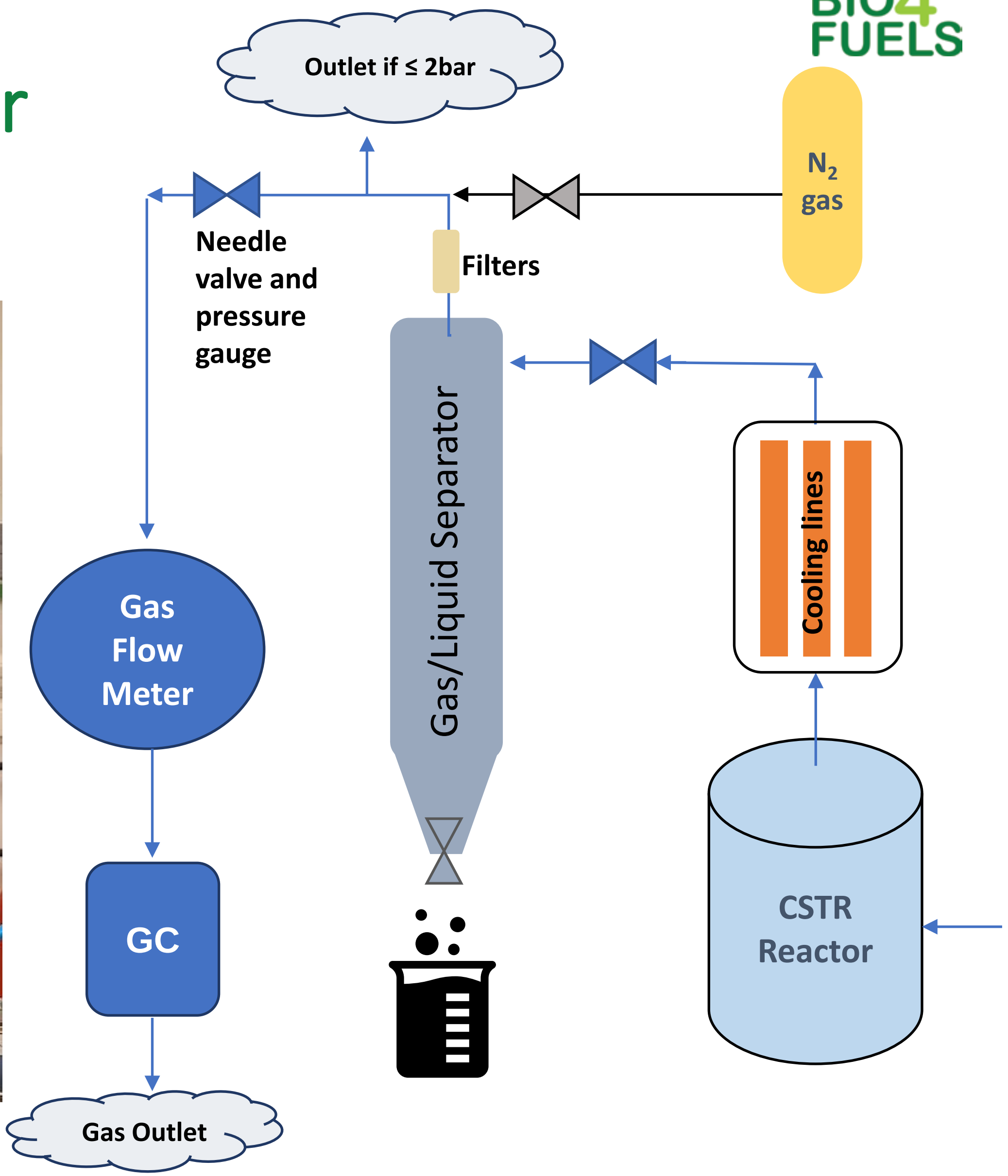
Reactor upgrades for gas detection for continuous operation

- New flow meter connected
- Installation of gas mixer system for option to use carrier gasses (e.g. Nitrogen)





Continuous Reactions Experimental Matrix

- Same conditions tested as for Batch
  - Pine and spruce
  - 350°C/ 400°C
  - Bark : 10%, 20%
- Additional test for stirring speed
- Tested mixed wood species

Oil from 400 °C, Continuous

Oil from 350°C, Continuous

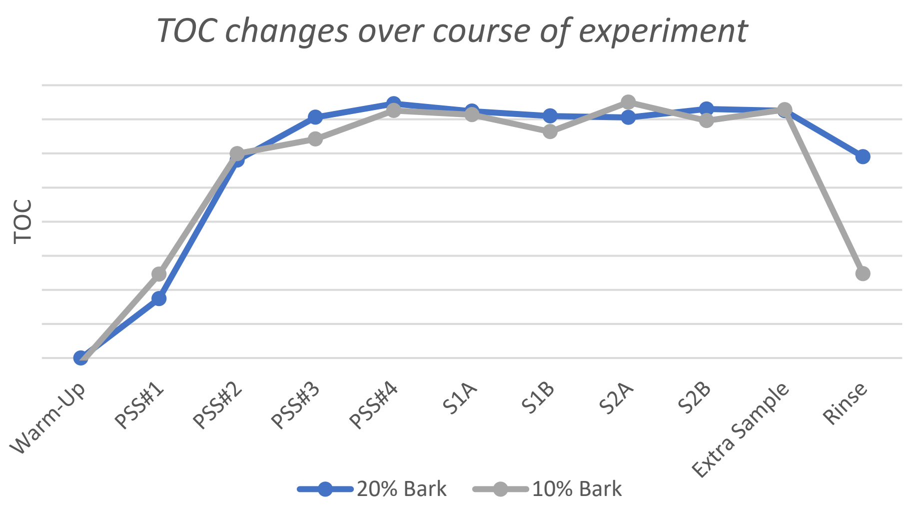
Hydrochar

- More char observed with bark.
- As mentioned, more char produced at lower speeds of stirring
- Slightly higher char at 400C than 350C
- Hydrochar produced was about 1-3% of biomass input by weight

Results removed – publication of data to come after completion of work

Total Organic Carbon – Aqueous Phase

- More carbon is converted to products in supercritical than subcritical conditions
- Second steady-state sample tends to have higher TOC
  - Possible lower conversion rate as char accumulates in reactor or,
  - More oil exiting system later in reaction, some of which is miscible in aqueous phase
- Over the course of the entire experiment, TOC seems stable once steady state is reached



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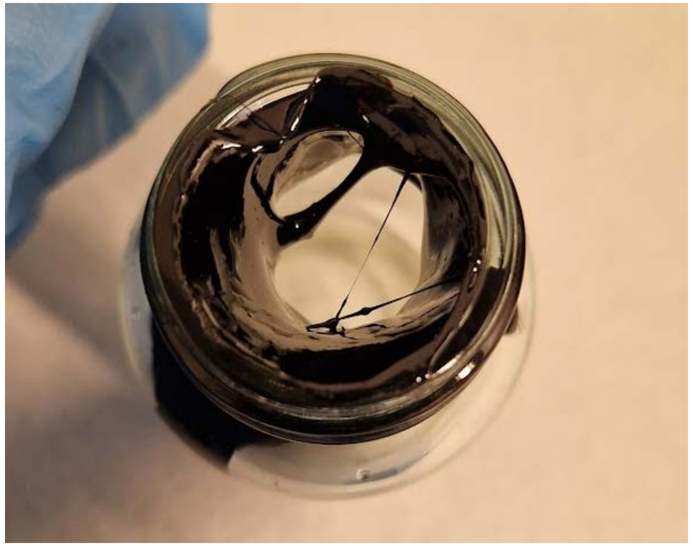
Bio-oil yields

- Analysis in progress for quality and energy values
- Yields are in alignment with literature (25-35% oil)



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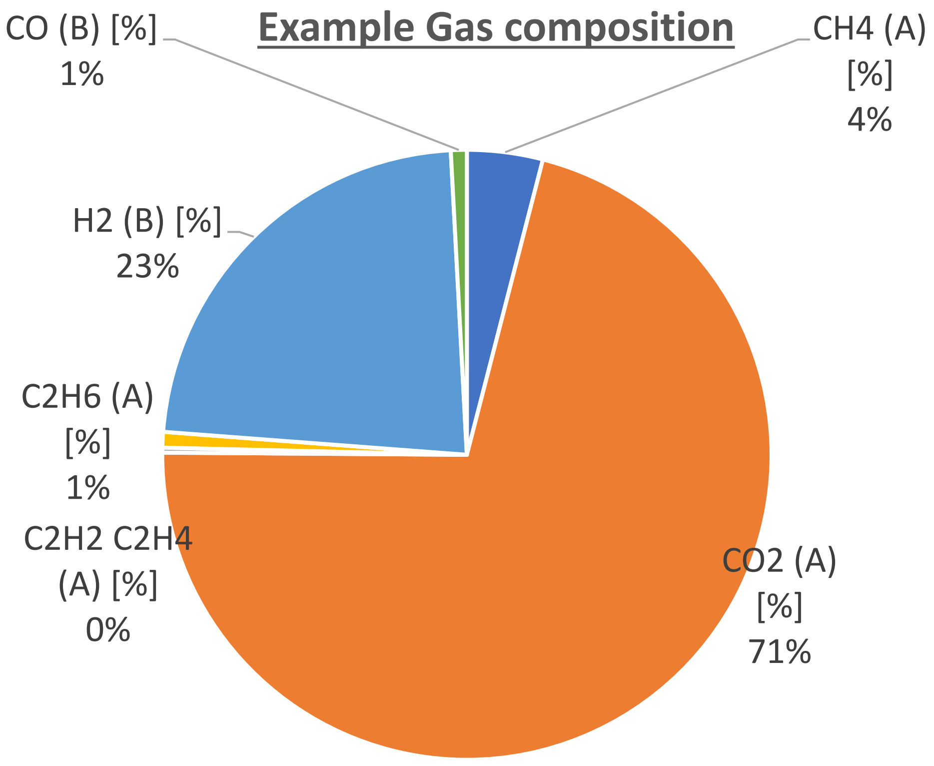
Gas Production

- More gas for samples with bark included
- Lower temperature produces less gasses
- Supercritical temperatures result in higher gas production



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Subcritical vs Supercritical Conditions

- Both spruce and pine have been tested at subcritical (350 C) and supercritical (400 C) conditions
- Prior to steady state, pH at outlet was slightly lower, increasing 1 pH unit during the 1.5h transition to steady state

At supercritical conditions

pH is higher

Slightly more char and less oil

More analysis is needed for further comparison (in progress)

Results removed – publication of data to come after completion of work

Type of analysis for products ordered

Test name test/standard, test method	Oil testing	Char testing	Aqueous testing
Determination of water content - Karl Fisher method	Q/LCA/75/B:2022 (Karl Fisher)	-	PN-EN ISO 18122:2023-05 (dry matter content)
Determination of ash content	ISO 8006 or PN-77/C-97065	PN-EN ISO 18122:2023-05 (550° C)	ITPE procedure (550° C)
Determination of C,H, N, S content	O}LCA/81/B:2022	PN-EN ISO 16948:2015-07 PN-EN ISO 16994:2016-10	PN-EN ISO 16948:2015-07 PN-EN ISO 16994:2016-10
Determination of carbon residue	Internal method	-	-
Determination of HHV	O}LCA/43/B:2022	PN-EN ISO 18125:2017-07	-
Determination of: S, Cl, Ca, K, P, Si, Na, Al, Mg, Zn, As, B, Ba, Cd, Co, Fe, Cr, Ni, Mo, Cu, Ga, Li, Mn, Mo, Ni, Pb, Sc, Se, Sr, Ti, V	ITPE procedure	ITPE procedure	ITPE procedure
Sample preparation	-	O}LCA/75/B:2022	-

Final Steps of the Project

- Completion/Repeat of continuous experiments
- Analysis of products and publication
- Summer student project 2024: Investigation of improved separation techniques

Ethyl Acetate
Diethyl ether
n-Heptane
Toluene with Brine
Acids/Bases
Changes in Density

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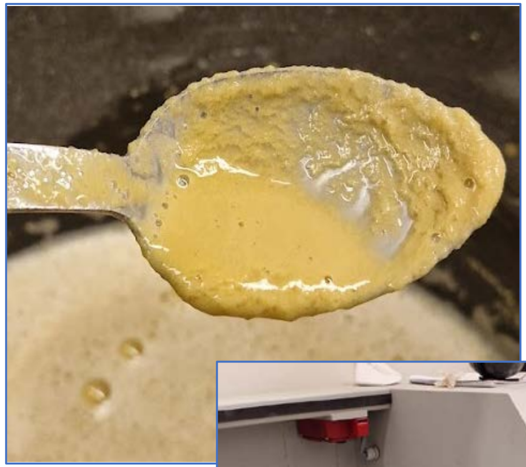
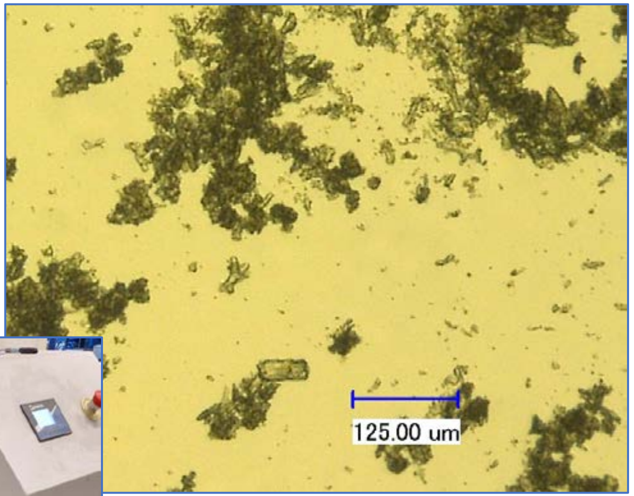
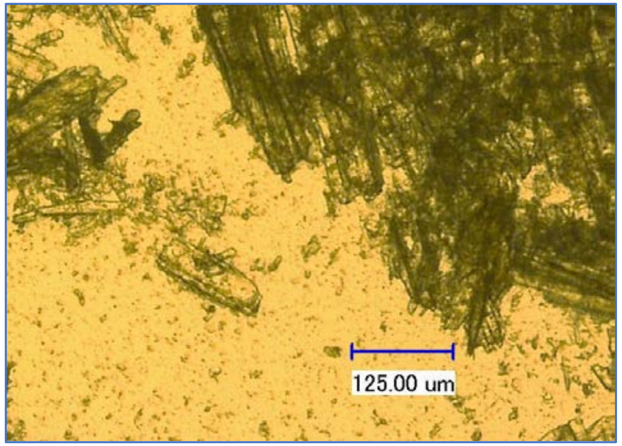
Hurdles: Leaks, clogs, milling optimization, pump failure, mill failure







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