

HTL-based liquid fuel production: First results from the European collaborative project HyFlexFuel

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Outline

- ***The HyFlexFuel project***
- ***First pilot-scale HTL campaign***
- ***Catalytic upgrading of biocrude***
- ***Anaerobic digestion of HTL process water***
- ***Conclusions***

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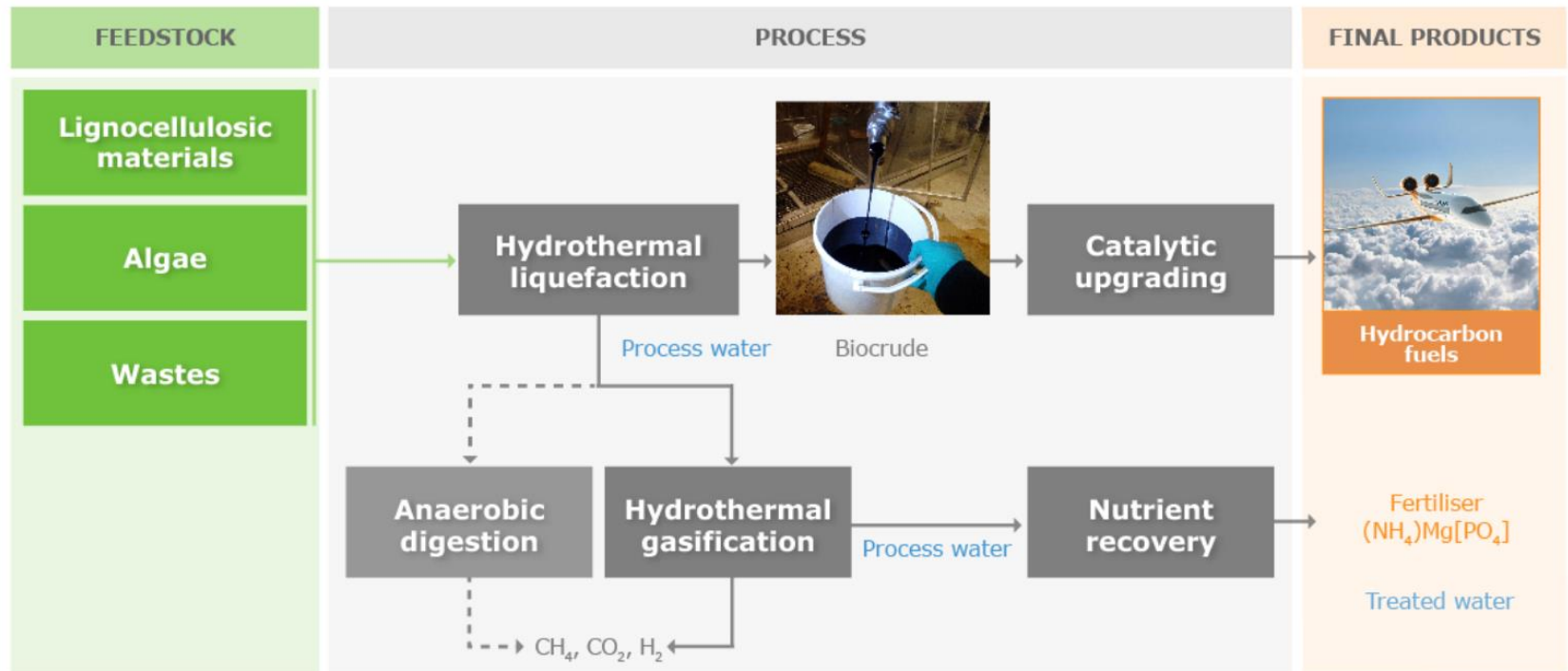
HyFlexFuel: Objectives

Development of a process chain to produce sustainable liquid fuels based on **hydrothermal liquefaction** of **various biomass feedstocks**

■ ***Specific objectives***

- **Demonstrate compatibility with diverse biomass feedstock portfolio (incl. algae and waste streams, such as sewage sludge)**
- **Increase energy and carbon efficiency through improved heat integration and product recovery**
- **Valorise organic and inorganic components in residual process streams**
- **Upgrade biocrude by catalytic hydrotreatment into fuel products and demonstrate their drop-in capability**
- **Assess technical, socio-economic and environmental performance potentials**

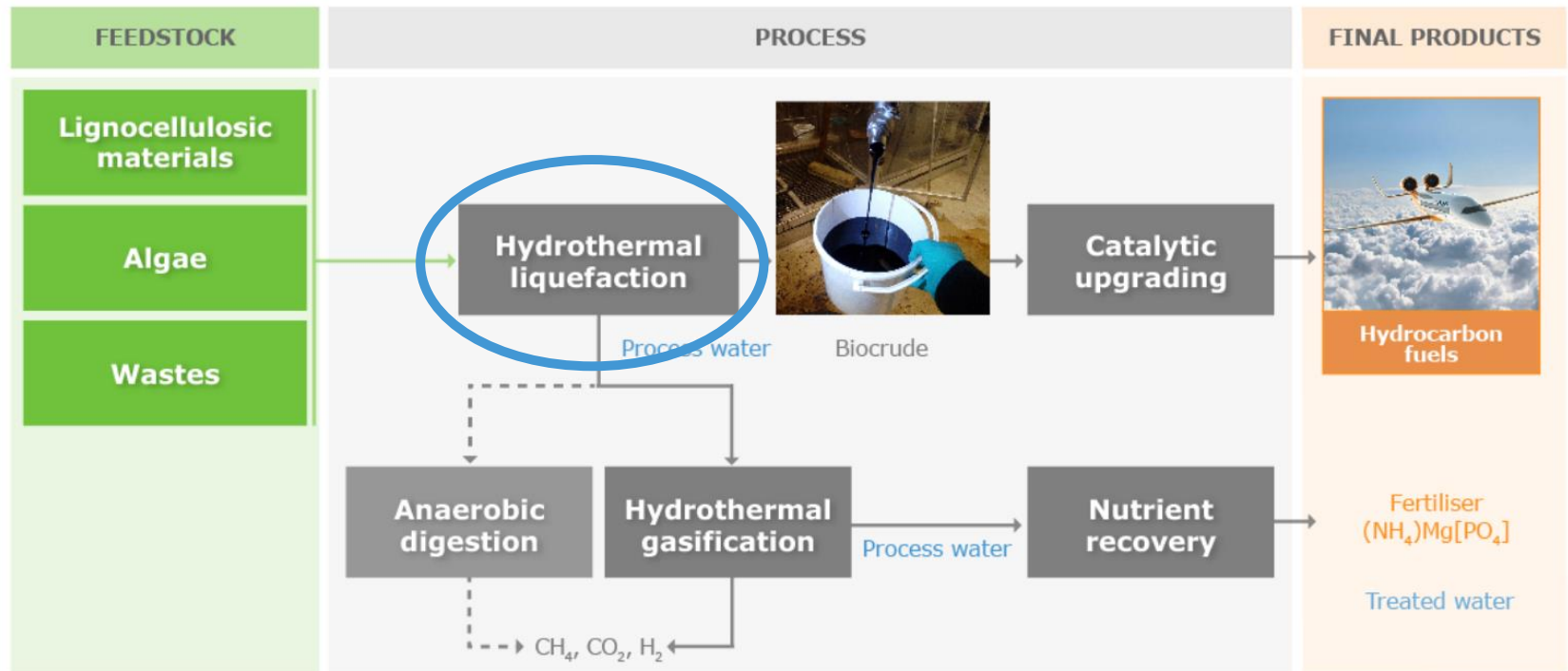
The HyFlexFuel process



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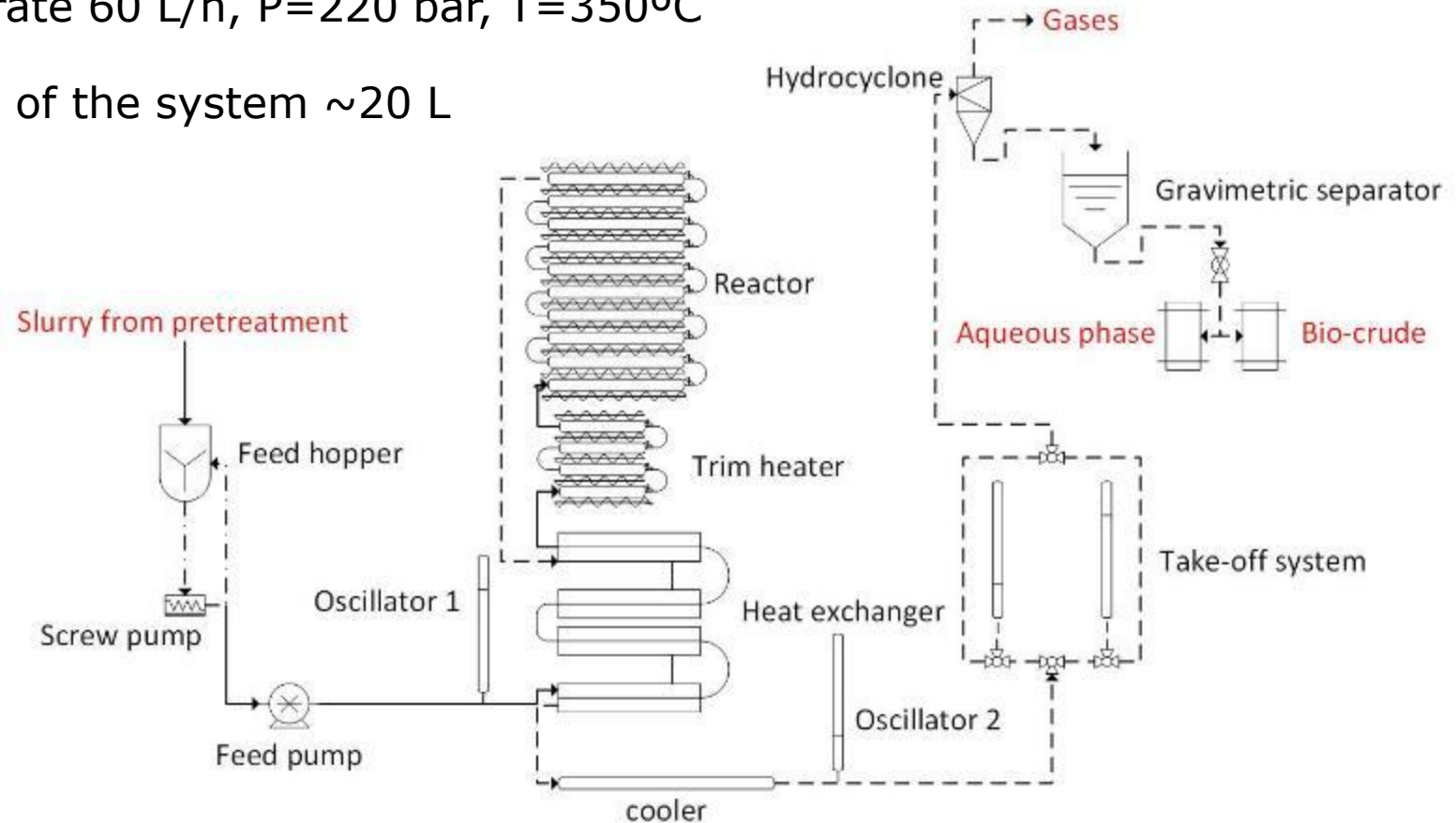


Pilot-scale HTL



Typical flow rate 60 L/h, $P=220$ bar, $T=350^{\circ}\text{C}$

Total Volume of the system ~ 20 L



First HTL campaign

■ Three „model feedstocks“

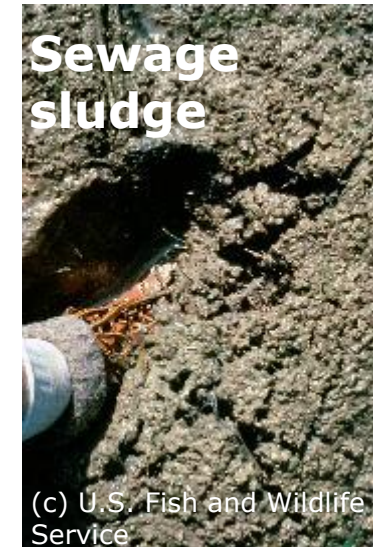


■ Input

- **Slurry: 250 kg (Miscanthus, Spirulina), 500 kg (sewage sludge)**

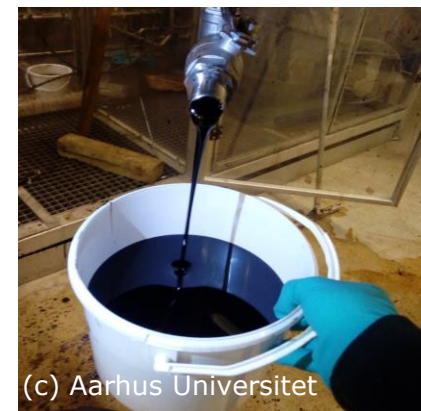
First HTL campaign

■ Three „model feedstocks“

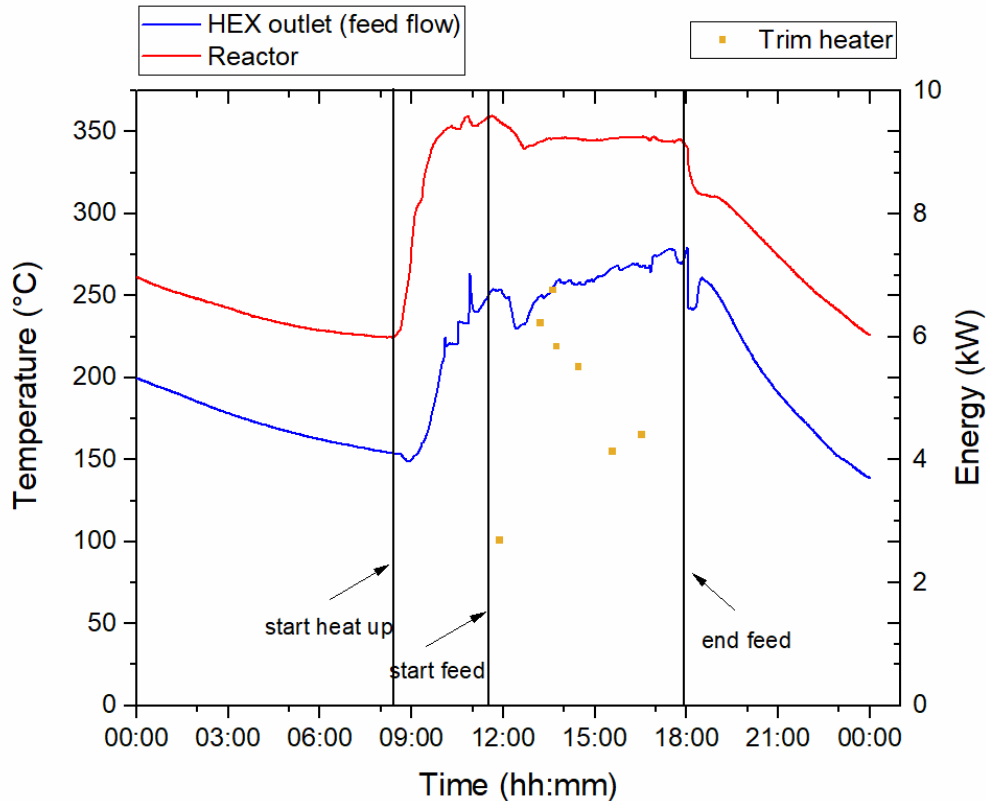


■ Output

- >10 kg biocrude and >200 L aqueous phase per feedstock



Heat recovery in heat exchanger



Time	HR (%)
12:00-13:00	69.1
13:00-14:00	73.3
14:00-15:00	74.8
15:00-16:00	76.7
16:00-17:00	77.6
17:00-18:00	79.9
Average	75.2

- Heat recovery increases over duration of experiments
- Longer run times should further enhance heat recovery (> 80%)

Anastasakis *et al.*, Assessing Hydrothermal Liquefaction of Lignocellulosic Biomass, Microalgae and Sewage Sludge at Pilot Scale, 26th European Biomass Conference & Exhibition, Copenhagen, 2018.

Process performance

Anastasakis et al., Assessing Hydrothermal Liquefaction of Lignocellulosic Biomass, Microalgae and Sewage Sludge at Pilot Scale, 26th European Biomass Conference & Exhibition, Copenhagen, 2018.

	Miscanthus	Spirulina	Sewage sludge
Flow rate (L/h)	60	60	60
DM content (%)	0.15	0.16	0.04
Time (h)	1	1	1
Feedstock consumed (kg, dry)	9	9.8	2.4
Energy in feedstock (kW, dry)	42.7 (HHV=17.1MJ/kg)	63.1 (HHV=23.1MJ/kg)	13.2 (HHV=19.8MJ/kg)
Bio-crude yield (wt.%)	26.2	32.9	24.5
Energy in bio-crude (kW, dry)	19.9 (HHV=30.6 MJ/kg)	32 (HHV=35.6 MJ/kg)	4.4 (HHV=26.8 MJ/kg)
η_{th} (%)	46.5	50.7	33.2
Trim heater energy req. (kW)	4.4	5.5	5.4
Reactor energy req. (kW)	2	2.8	2.5
Main pump energy req. (kW)	0.7	0.7	0.7
η_{tot} (%)	39.9	44.4	20.1

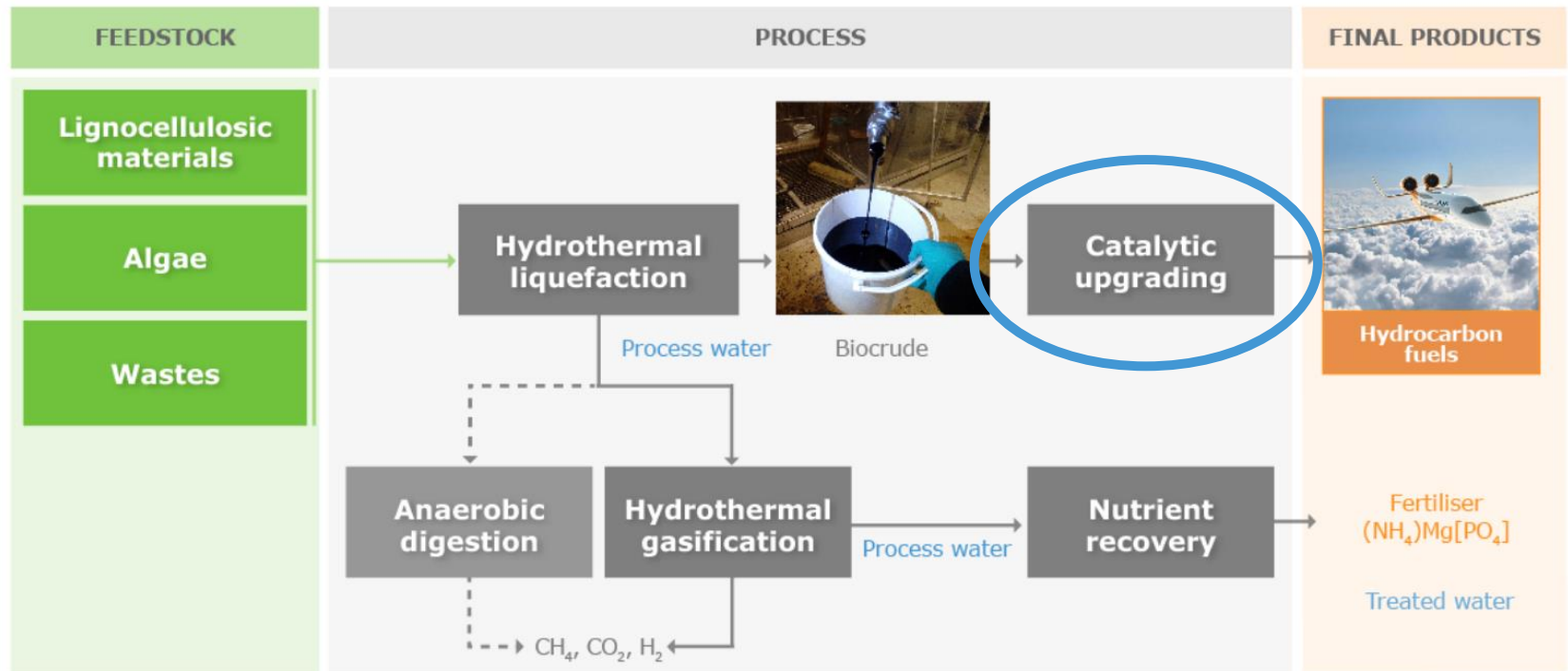
HTL campaign: Summary & outlook

- ***Successful liquefaction of three different feedstocks***
 - Samples (biocrude, aq. phase, solids) could be supplied to partners
 - Heat recovery of up to 80%
 - Average biocrude yields 26.2% (Miscanthus), 32.9% (Spirulina) and 24.5% (sewage sludge)
- ***Further work will focus on improvement of process conditions (e.g. heat recovery, in-line filtration) and other feedstocks***

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The HyFlexFuel process



First catalytic upgrading experiments

- **Catalytic hydrotreatment of biocrudes from first HTL campaign**
 - **Spirulina**, sewage sludge, miscanthus
 - Batch mode
 - Screening of reaction conditions and pre-treatment procedures



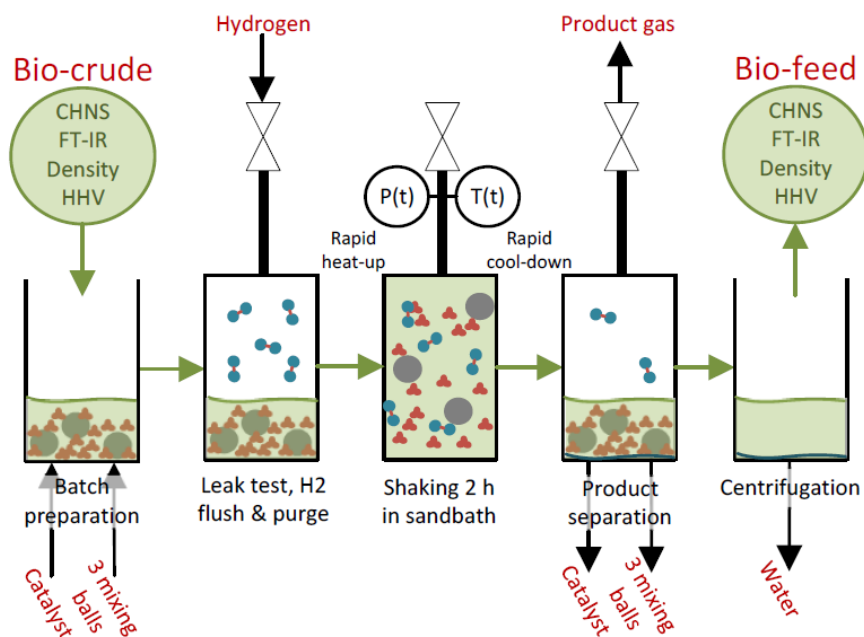
- **Objectives**
 - To identify sample-specific challenges
 - To find suitable reaction conditions for upgrading campaigns in **continuous mode**
 - To collect data enabling specific catalyst design



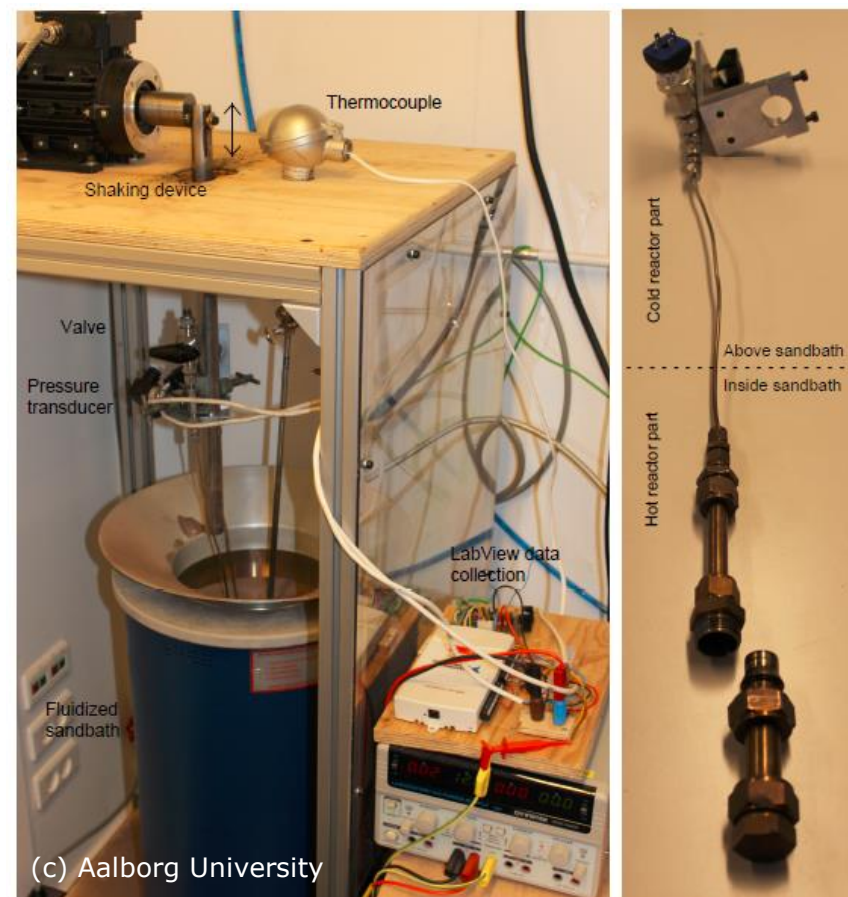
(c) Aalborg University
Spirulina biocrude

First catalytic upgrading experiments

■ Experimental setup

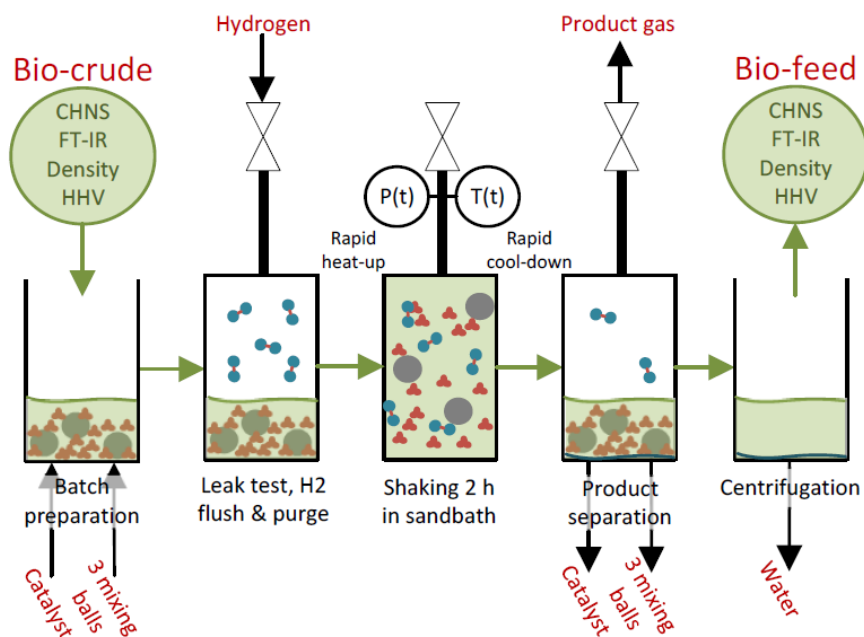


Jensen et al., *Fuel* **2016**, 165, 536-543.



First catalytic upgrading experiments

■ Experimental setup



Jensen et al., *Fuel* **2016**, 165, 536-543.

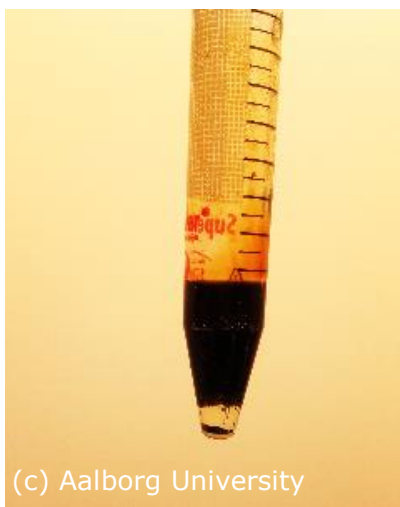
- Biocrude 4 g, Catalyst 2 g
- Commercial NiMo/Al₂O₃ catalyst
- Pre-sulfided

Reaction condition	T (°C)	P ₀ (bar)	t (h)
Mild	250	40	2
Medium	300	60	3
Severe	300	80	4



Catalytic upgrading of *Spirulina* biocrude

	Dry ash free (wt. %)				H/C (-)	de-O (wt. %)	de-N (wt. %)
	C	H	N	O			
<i>Spirulina</i>	53.5	7.2	12.6	26.6	1.62	-	-
Biocrude	78.1	10.4	8.0	3.5	1.60	86.8	36.7
Mild	79.2	10.8	7.4	2.6	1.63	90.2	41.2
Medium	79.7	11.7	6.3	2.3	1.76	91.4	50.1
Severe	81.0	12.1	6.0	1.0	1.79	96.4	52.7

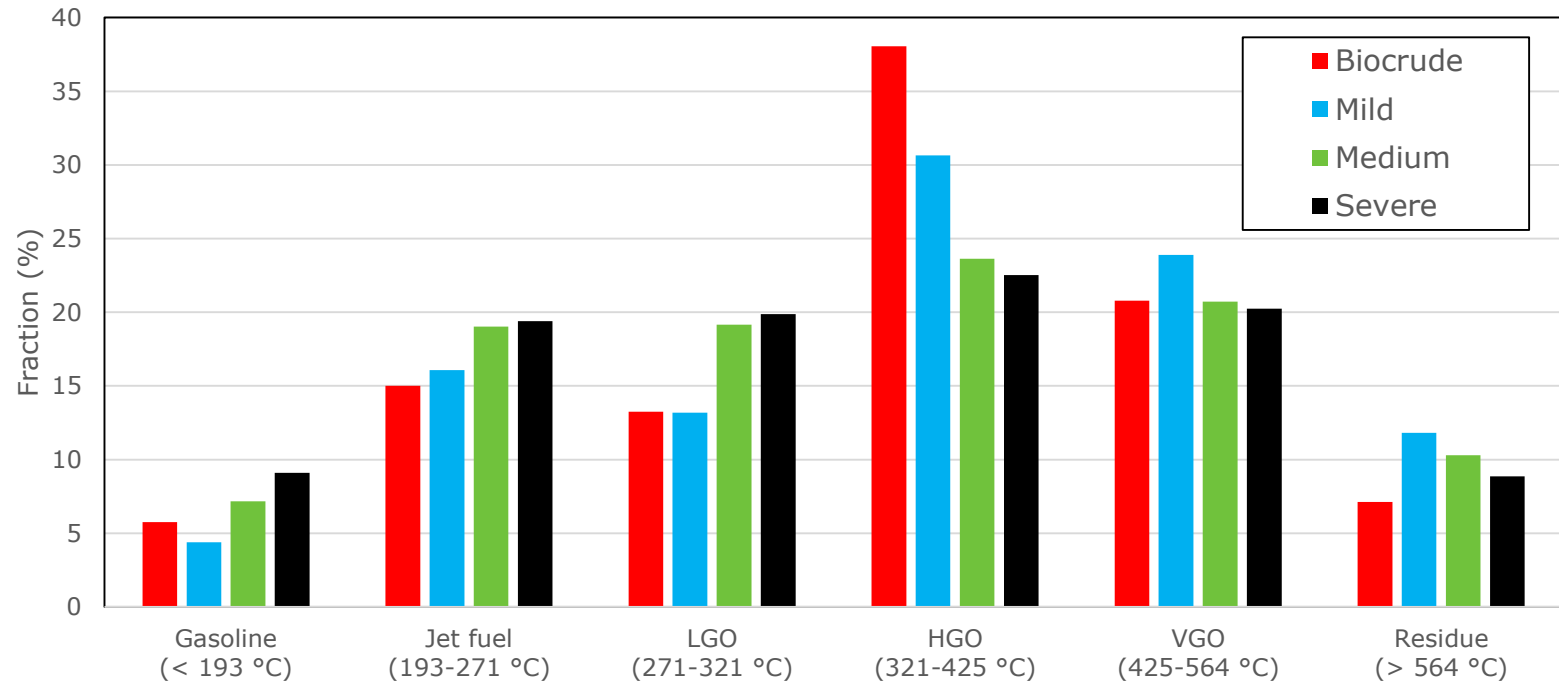


- More severe conditions result in more effective removal of heteroatoms
- Increasing H/C ratio
- Relatively high deoxygenation
- Denitrogenation is around or below 50%

(c) Aalborg University

Catalytic upgrading of *Spirulina* biocrude

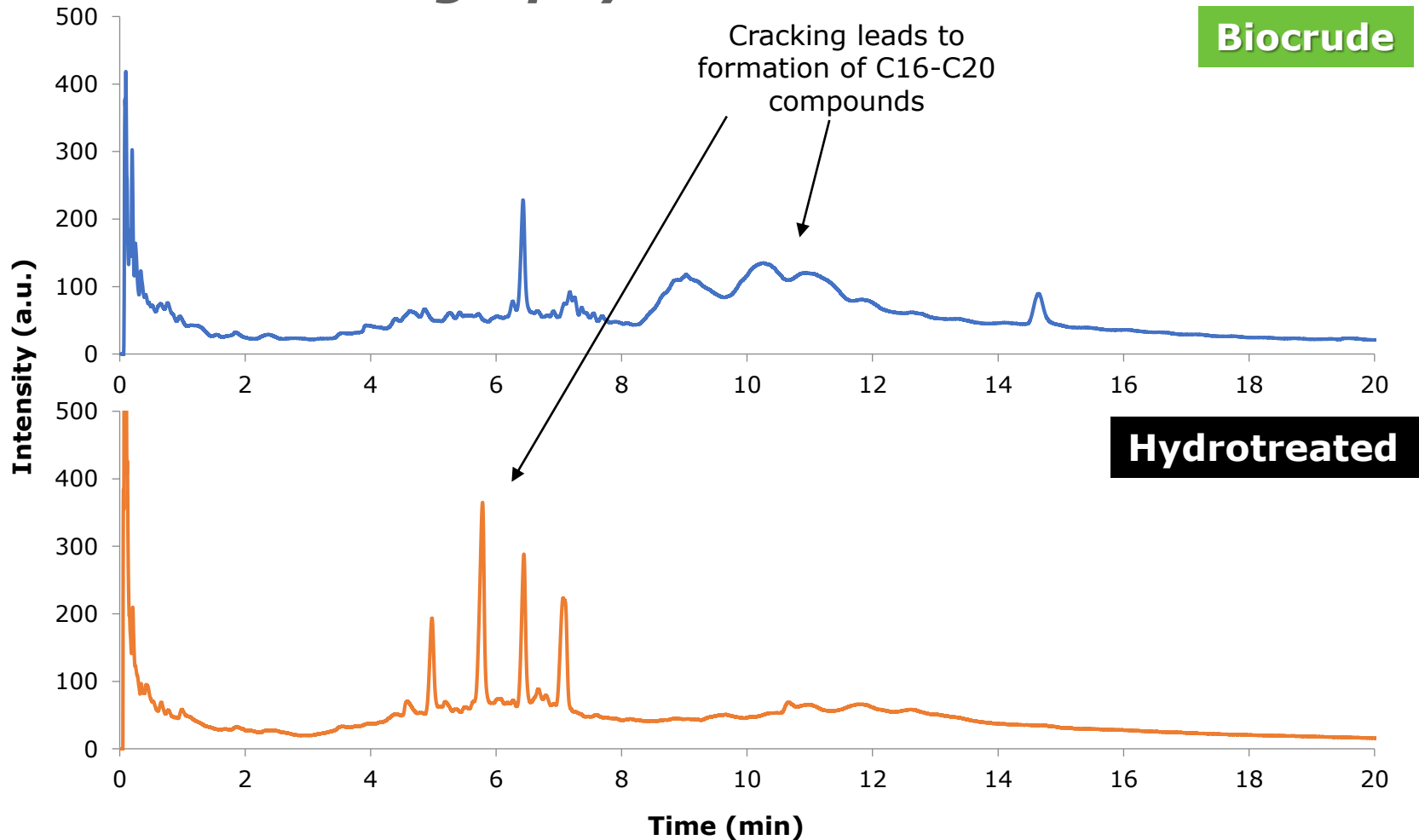
■ *Simulated distillation*



- **Simulated distillation according to ASTM D7169**
- **Increase in the lighter fractions, reduction of HGO**

Catalytic upgrading of *Spirulina* biocrude

■ Gas chromatography



Catalytic upgrading: Summary & outlook

- ***First batch experiments on catalytic upgrading of HTL biocrudes conducted***
- ***Fuel quality (H/C, de-O, de-N) substantially enhanced; de-N not yet sufficient***
- ***Gasoline and middle distillate fraction increased***

- ***Next steps***
 - More screening experiments
 - Improvement of pre-treatment techniques
 - Tailored catalysts
 - First experiments in continuous mode

HALDOR TOPSØE 

Catalytic upgrading: Summary & outlook

- **First batch experiments on catalytic upgrading of HTL biocrudes conducted**

- **Fu
en**

- **Ga
inc**

More details and data from catalytic upgrading experiments in HyFlexFuel soon to be presented by D. Castello *et al.* on the **7th International Symposium on Energy from Biomass and Wastes**, Oct. 15-18, 2018, Venice

- **Next steps**

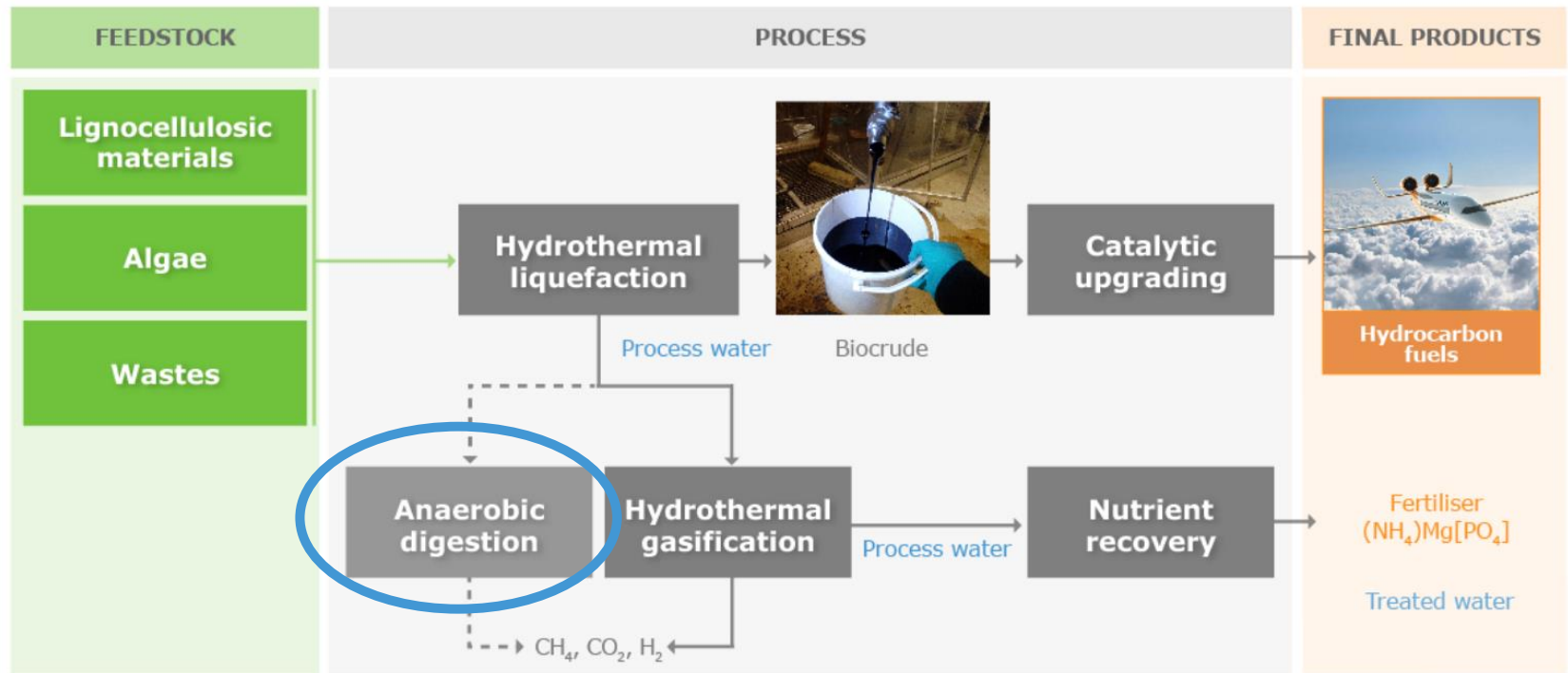
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Anaerobic digestion of HTL process water

■ *Biogas production potential test*



■ *Goal:*

- Determine max potential
- Detect acute toxicity/deficiency



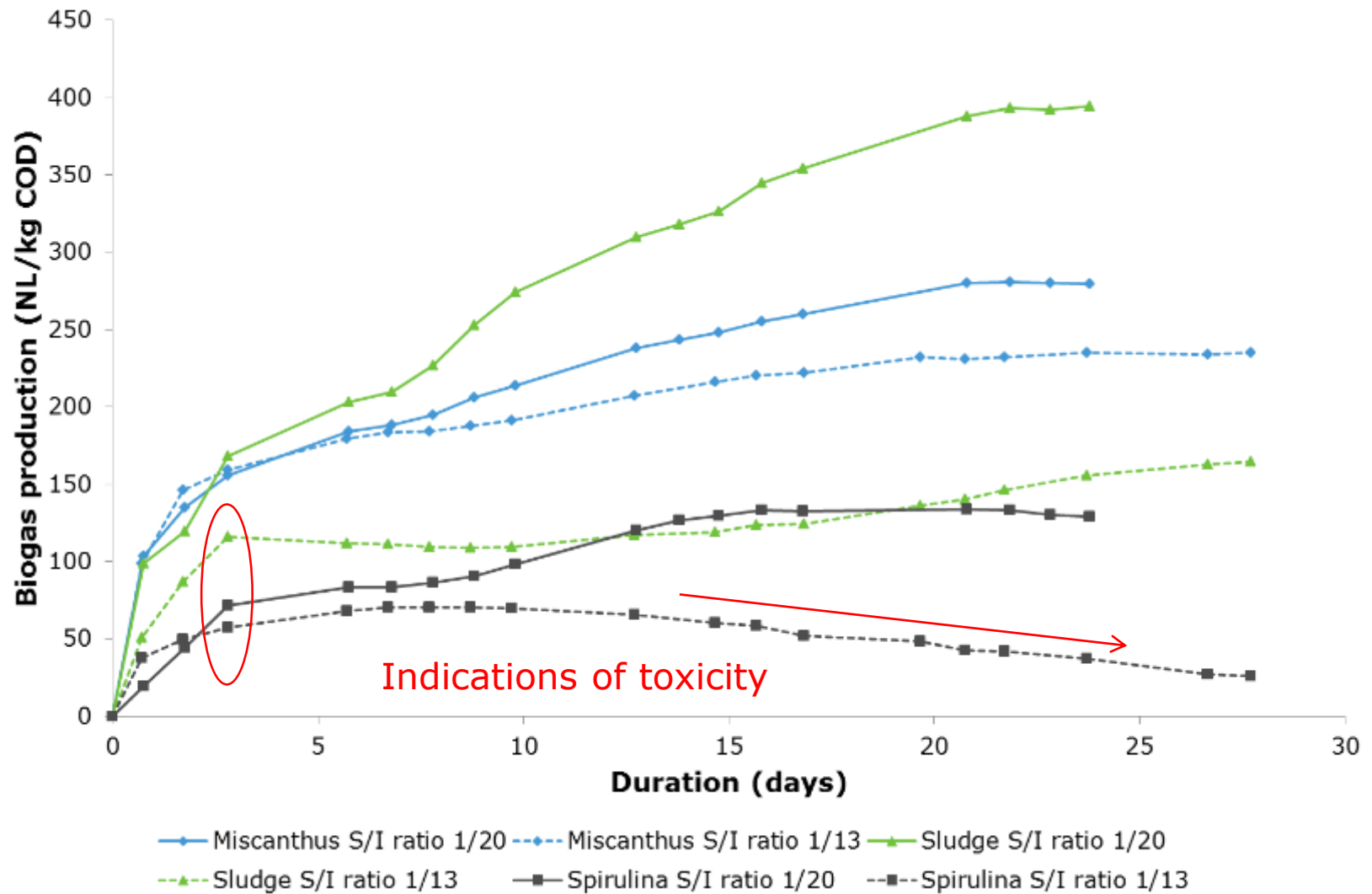
■ *Experimental setup*

- 10-100 g of substrate to 1 kg active inoculum
- Typical duration: 14 days or until daily gas production < 1% of cumulative production

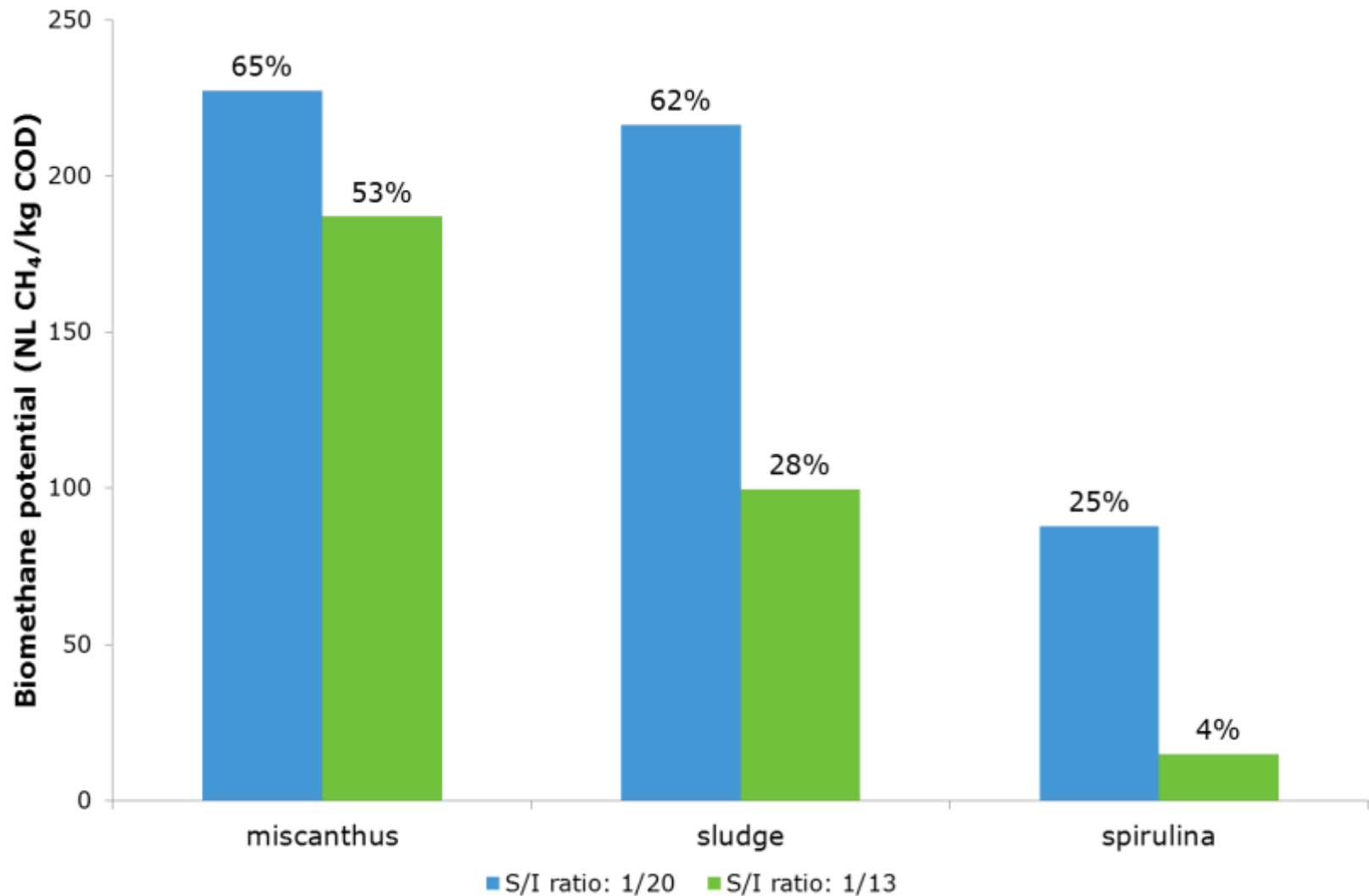


■ *Basis for continuous test set-up*

Anaerobic digestion of HTL process water



Anaerobic digestion of HTL process water



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Conclusions

- *HyFlexFuel develops a process chain for production of liquid fuels based on **hydrothermal liquefaction***
- *First **HTL campaign** conducted*
 - Successful liquefaction of Spirulina, sewage sludge, Miscanthus
 - Pilot-scale, relevant process conditions
- *First batch experiments on **catalytic upgrading** of HTL biocrudes conducted*
 - Deoxygenation successful; denitrogenation challenging
- ***Anaerobic digestion** of HTL process water*
 - Substantial methane formation, but also indication of toxicity observed

Thank you!

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