Hydrothermal pretreatment of difficult-to-digest biomass: Influence of pH, optimization of processing parameters and pilot plant operation

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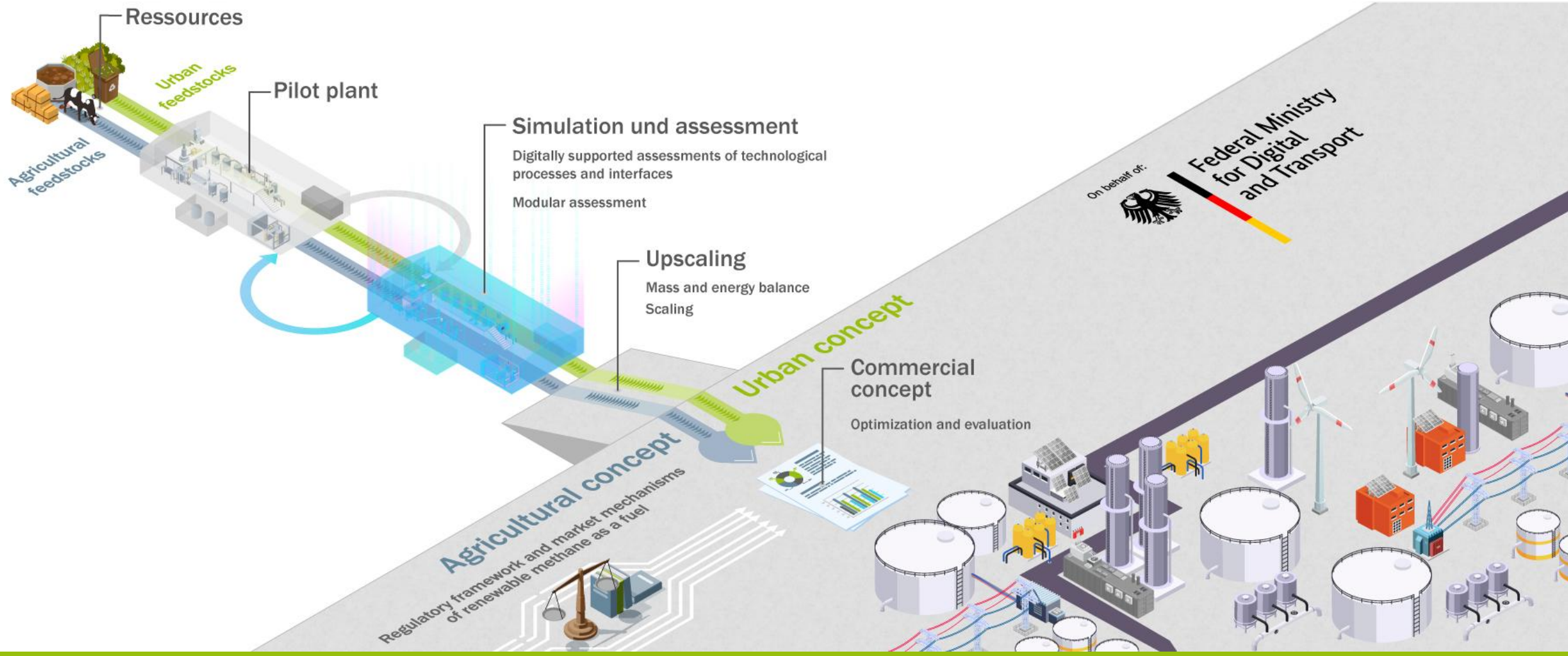
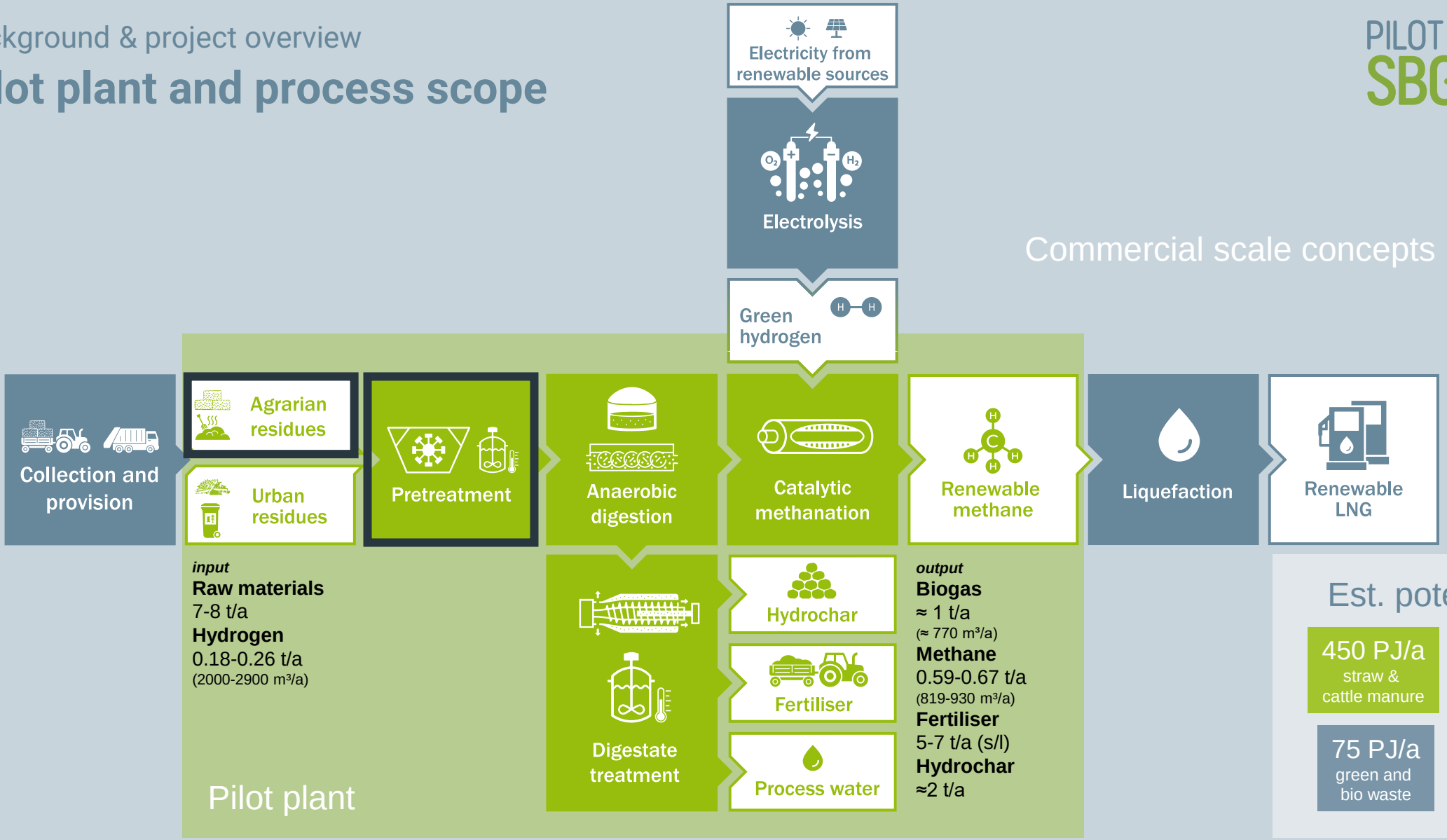
From pilot to commercial scale concepts

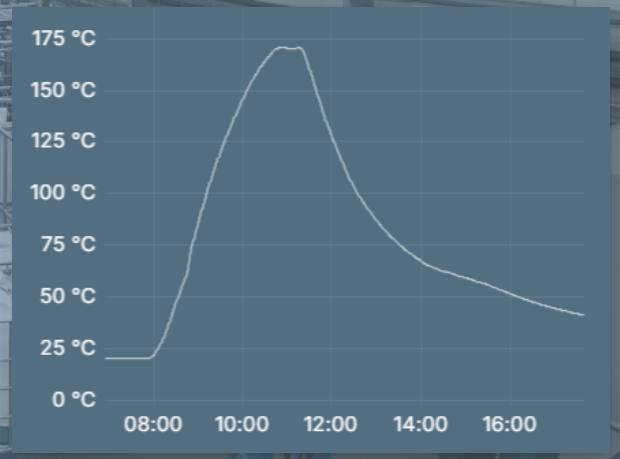


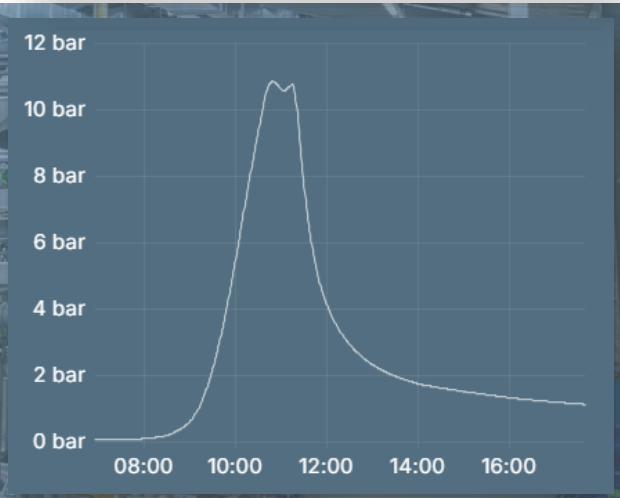
Pilot plant and process scope



m³ - standard cubic metre; Rough approximation of the mass balance. Biogas, fertiliser and hydrochar amount and composition depend on the raw material origin and quality

Source: [1]Naumann, K. et al. (2026): Fokusheft Standortanalyse und Ressourcenverteilung für erneuerbares LNG im Verkehr (in preparation)





Hydrothermal (pre-)treatment

Introduction

- Hydrothermal pre-treatment to enhance the digestibility of lignocellulosic biomass
- The main issue: the lignocellulosic matrix
  - Cellulose – crystalline structure slows microbial degradation
  - Hemicellulose – more accessible
  - Lignin – barely digestable anaerobically and physically shields the other two

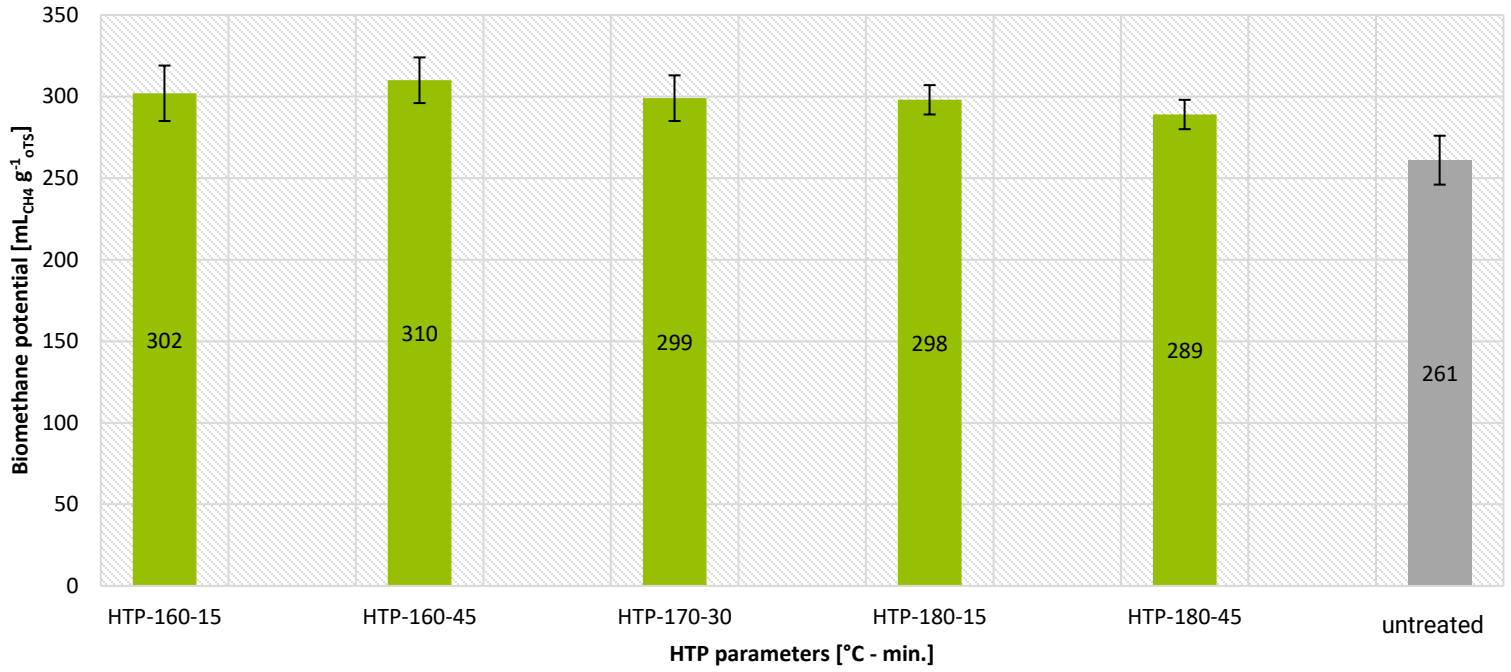
➢ HTP to thermochemically crack the matrix and start hydrolysis

➢ Improve kinetics and increase methane yield

➢ Sterilize the biomass

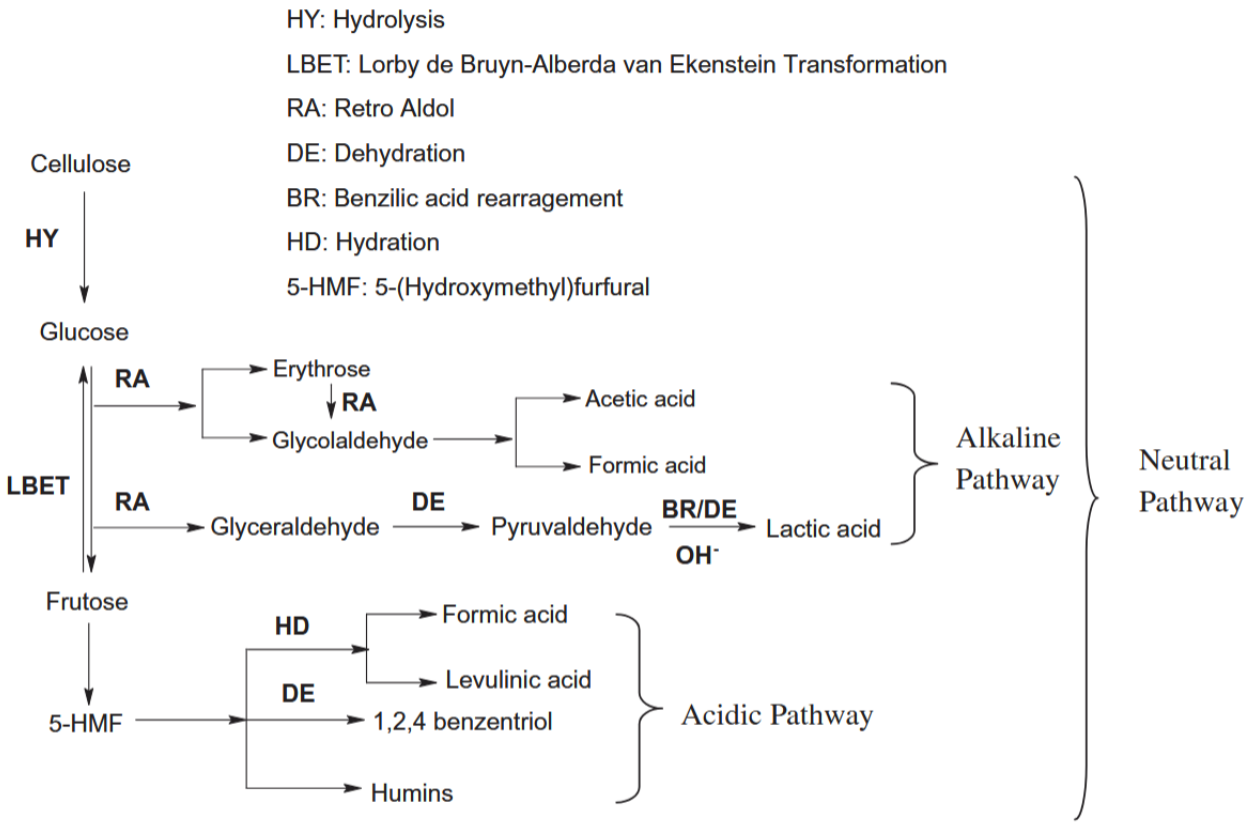
Previous results with straw/manure

- For a mixture of straw/manure we observed:
  - A significantly higher methane yield
  - HTP parameters with negligible influence
  - No accumulation of inhibitors



Hydrothermal chemistry

- Acidic conditions favor dehydration, which leads to 5-HMF/Furfural
  - ➢ Known inhibitors in AD
- Basic conditions favor retro aldol reaction, which leads to acetic/formic acid.
  - ➢ Methane precursors



Yin, S.; Tan, Z. Hydrothermal liquefaction of cellulose to bio-oil under acidic, neutral and alkaline conditions. *Applied Energy* **2012,** *92*, 234–239. DOI: 10.1016/j.apenergy.2011.10.041.

Materials & Methods

- Selected biomass:

  Oat husks, spelt husks, mixed leaves and bark mulch




Materials & Methods

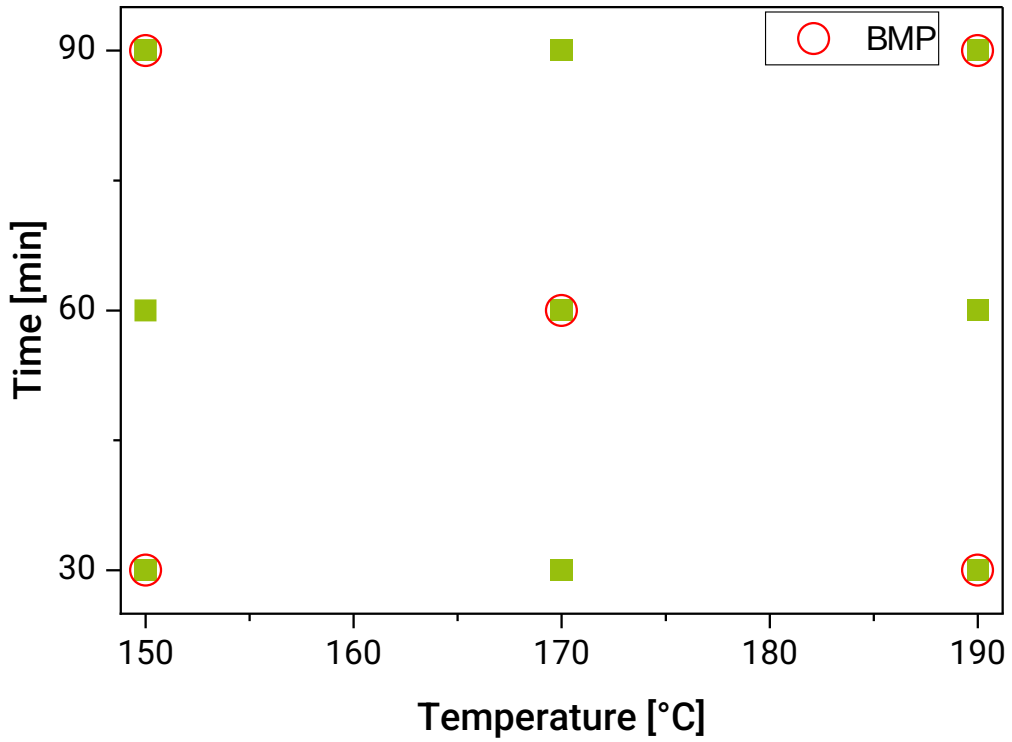
- Selected biomass:

  Oat husks, spelt husks, mixed leaves and bark mulch
- Experiments in 0.02 L and 0.5 L scale
- Analyses
  - HPLC-DAD-RID: Sugars, phenols, Furfural, 5-HMF
  - GC-MS: Organic acids
  - COD/TOC
  - CHNS

Experimental plan

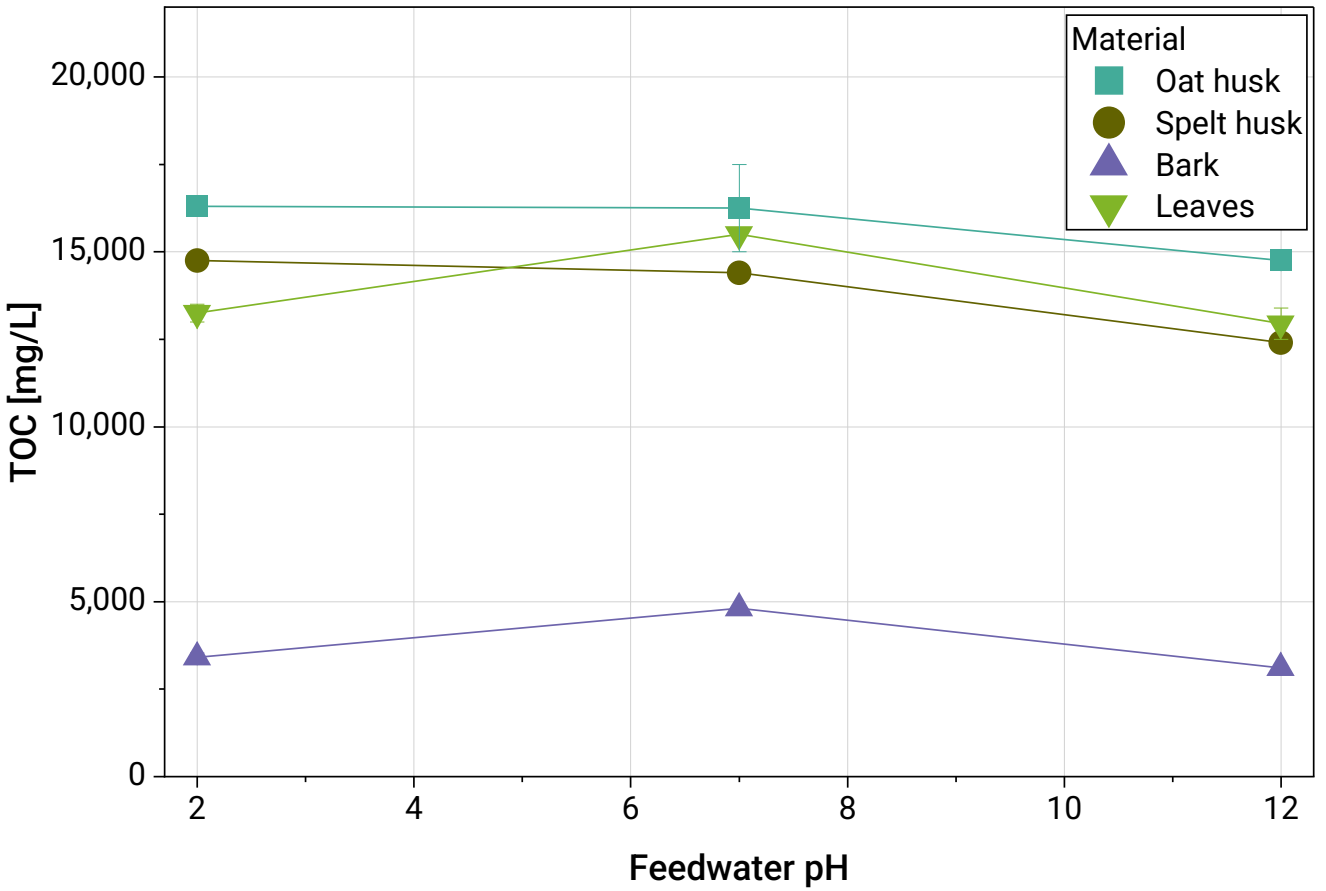
1) Variation of pH
   - Feedwater pH 2, 7, 12
   - HTP @ 170 °C for 60 min
   - BMP tests for lowest inhibitor concentration

2) Variation temperature and time
   - Optimize digestibility



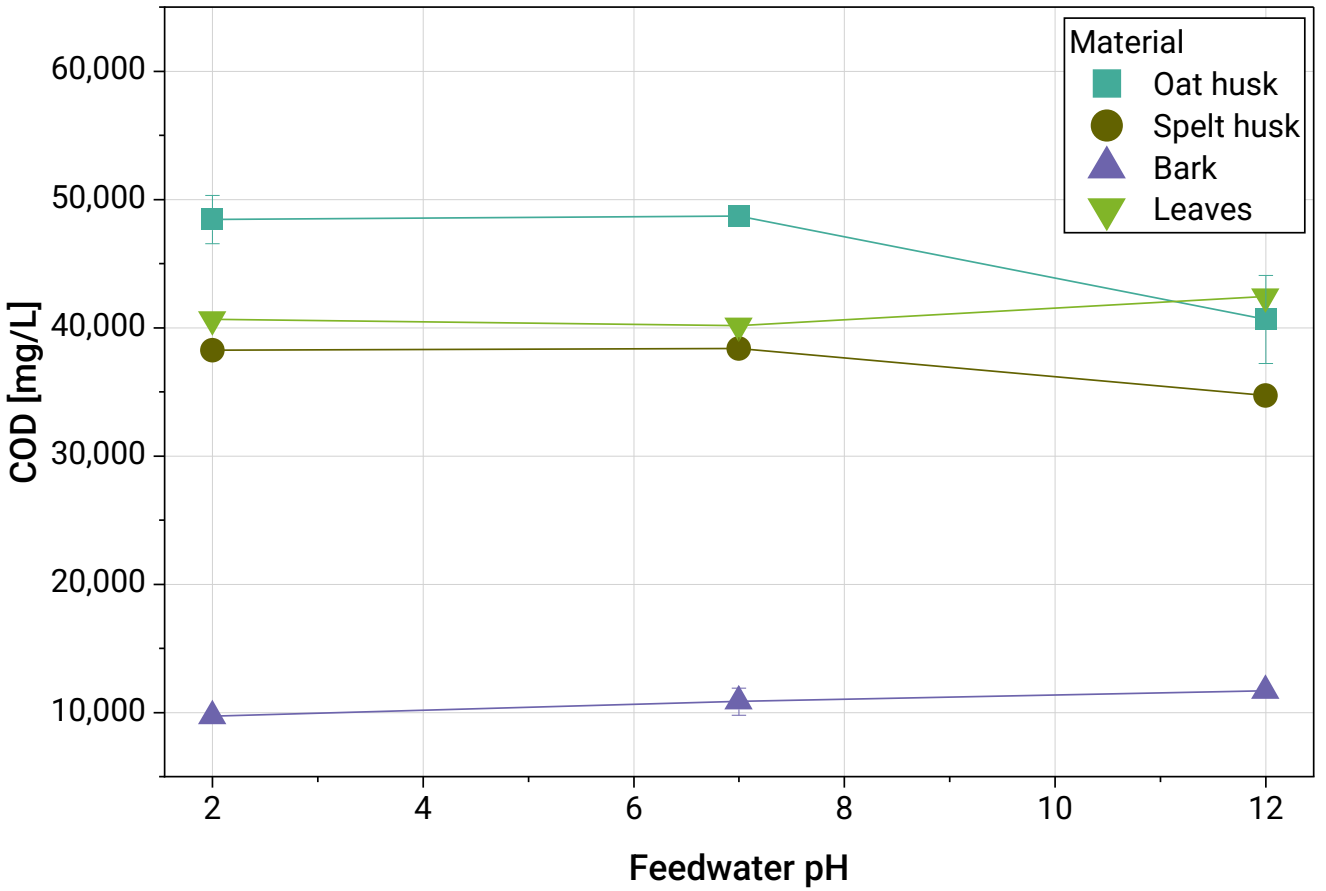
Influence of pH on TOC

- Slight decrease of TOC for the husks
- Maximum at pH 7 for bark and leaves
- Husks and leaves in a comparable range
- AP from bark has the lowest TOC, which suggests low degradation



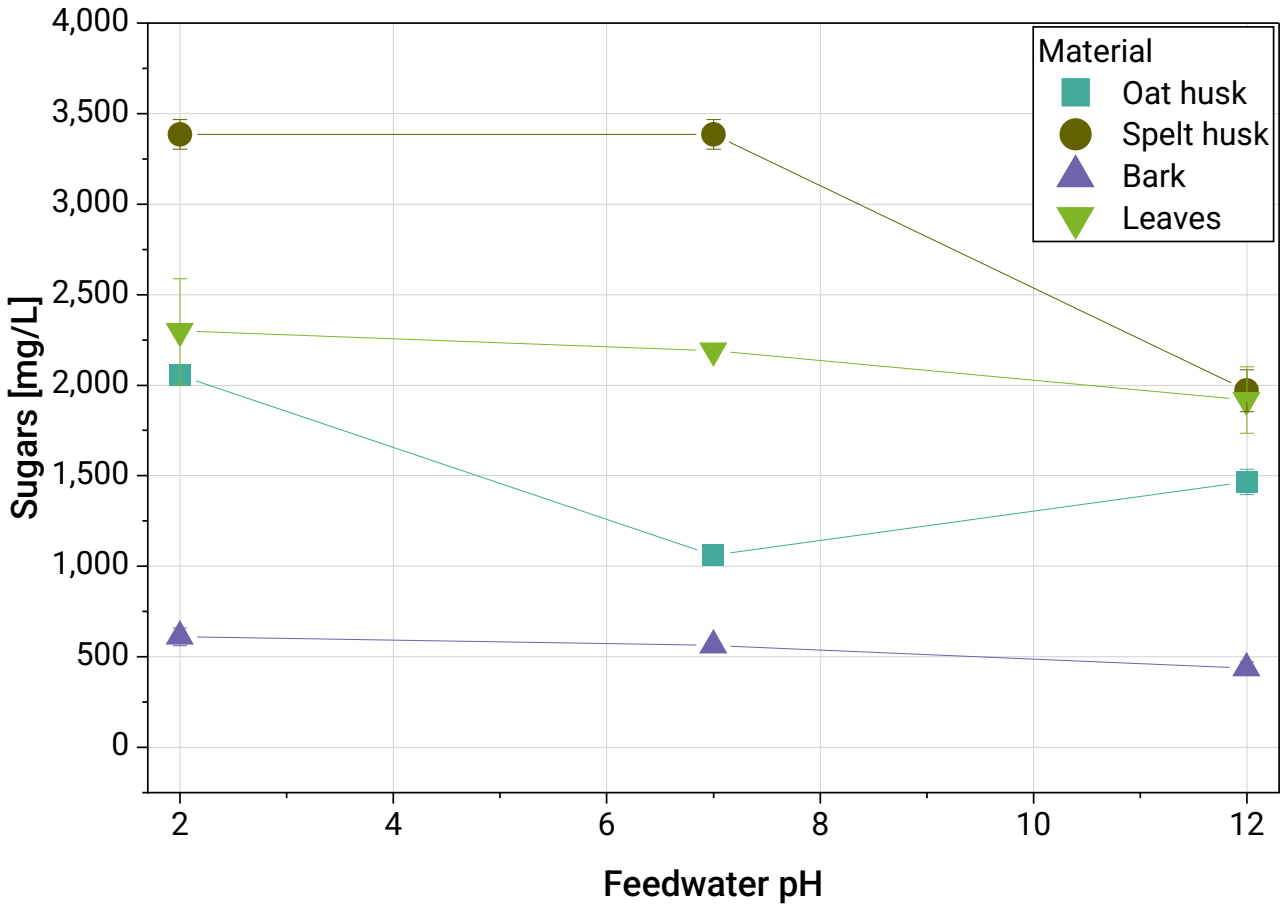
Influence of pH

- Trends similar to TOC
- COD : TOC ≈ 3:1



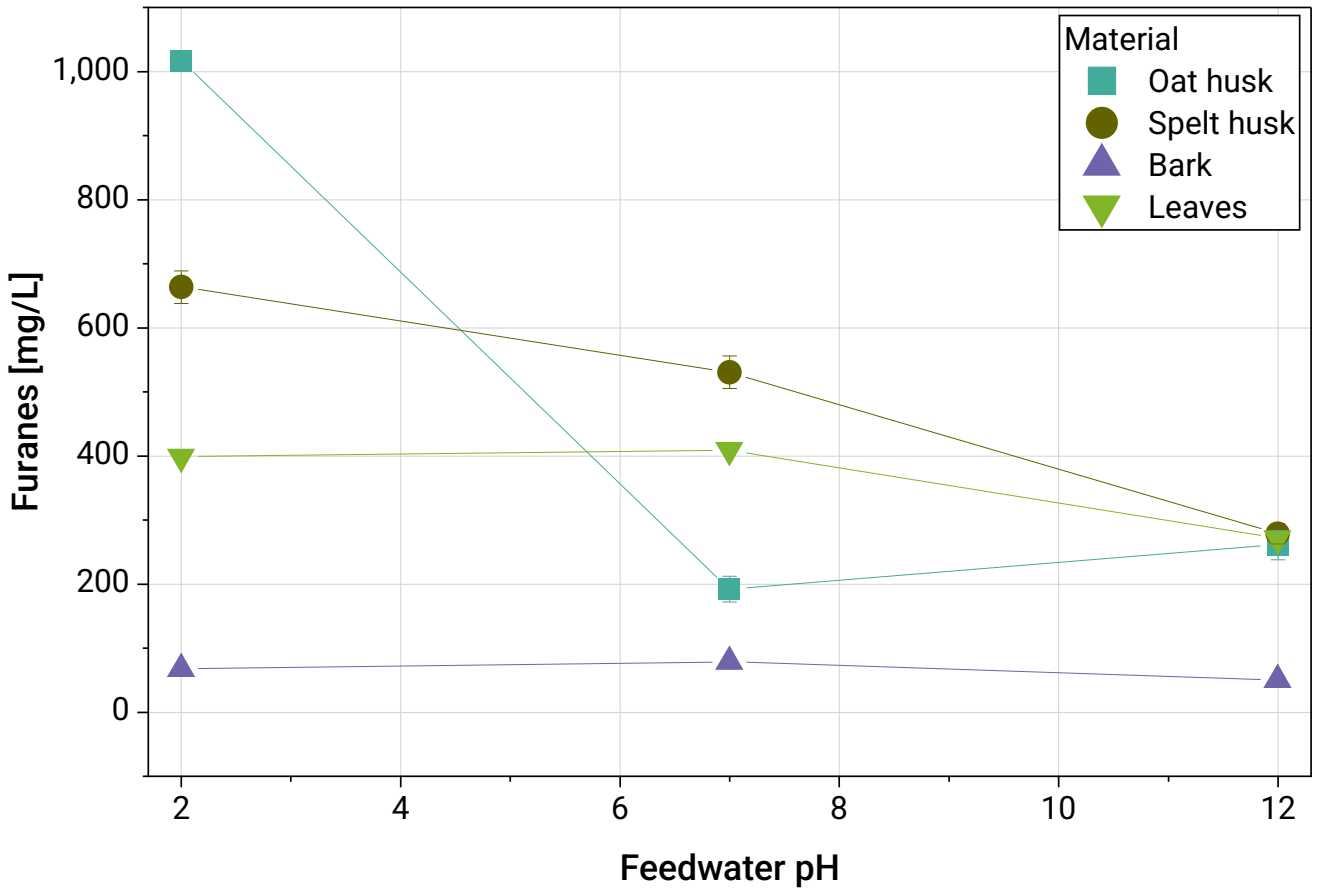
Influence of pH on sugars

- Sugar concentration decreasing for increasing feed water pH
  - Cellulose hydroylzes more efficiently in acidic conditions



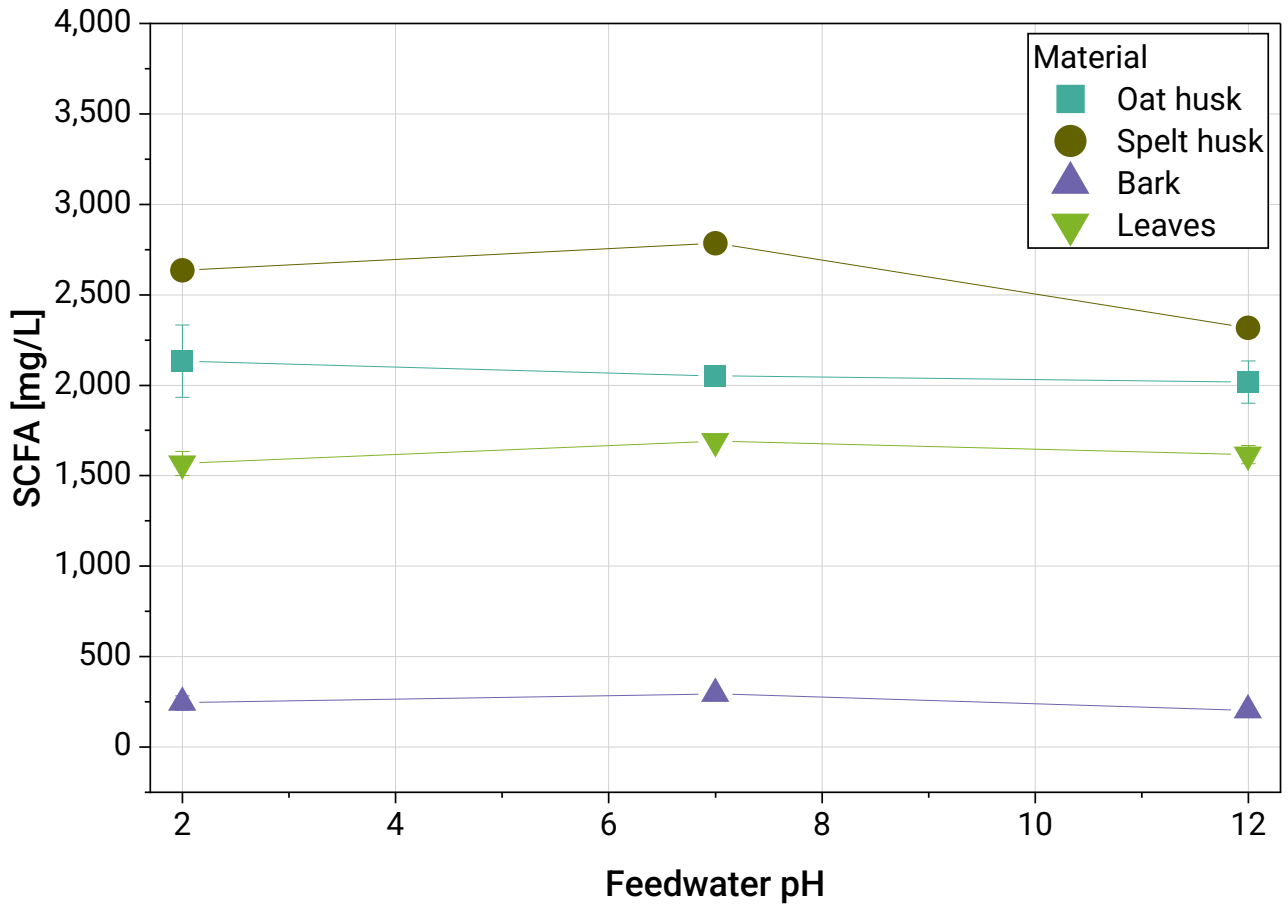
Influence of pH on furanics

- Furanics decreasing for increasing pH
  - Less dehydration towards 5-HMF



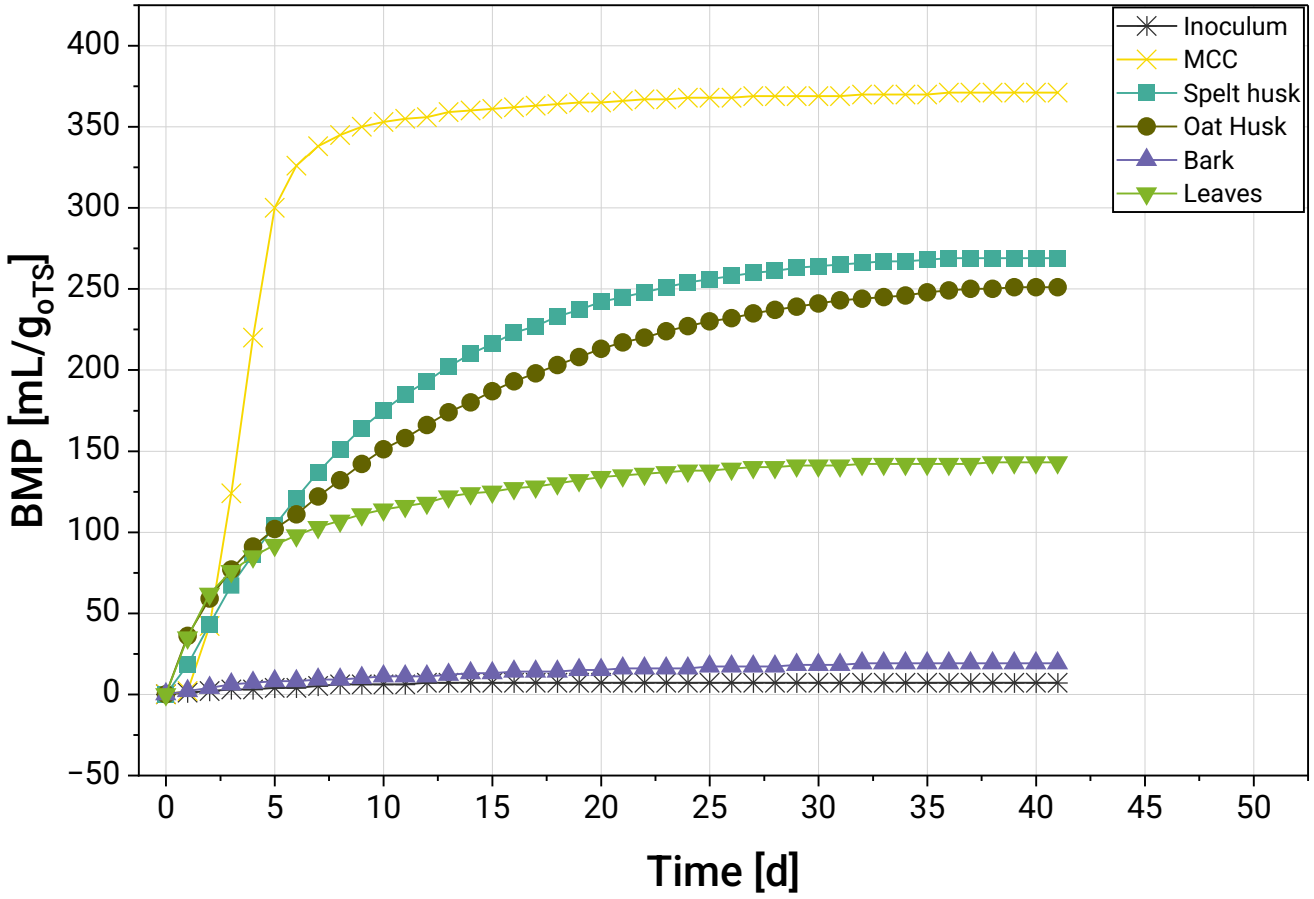
Influence of pH on furanics

- Effect of pH on SCFA less pronounced



Biological methane potential

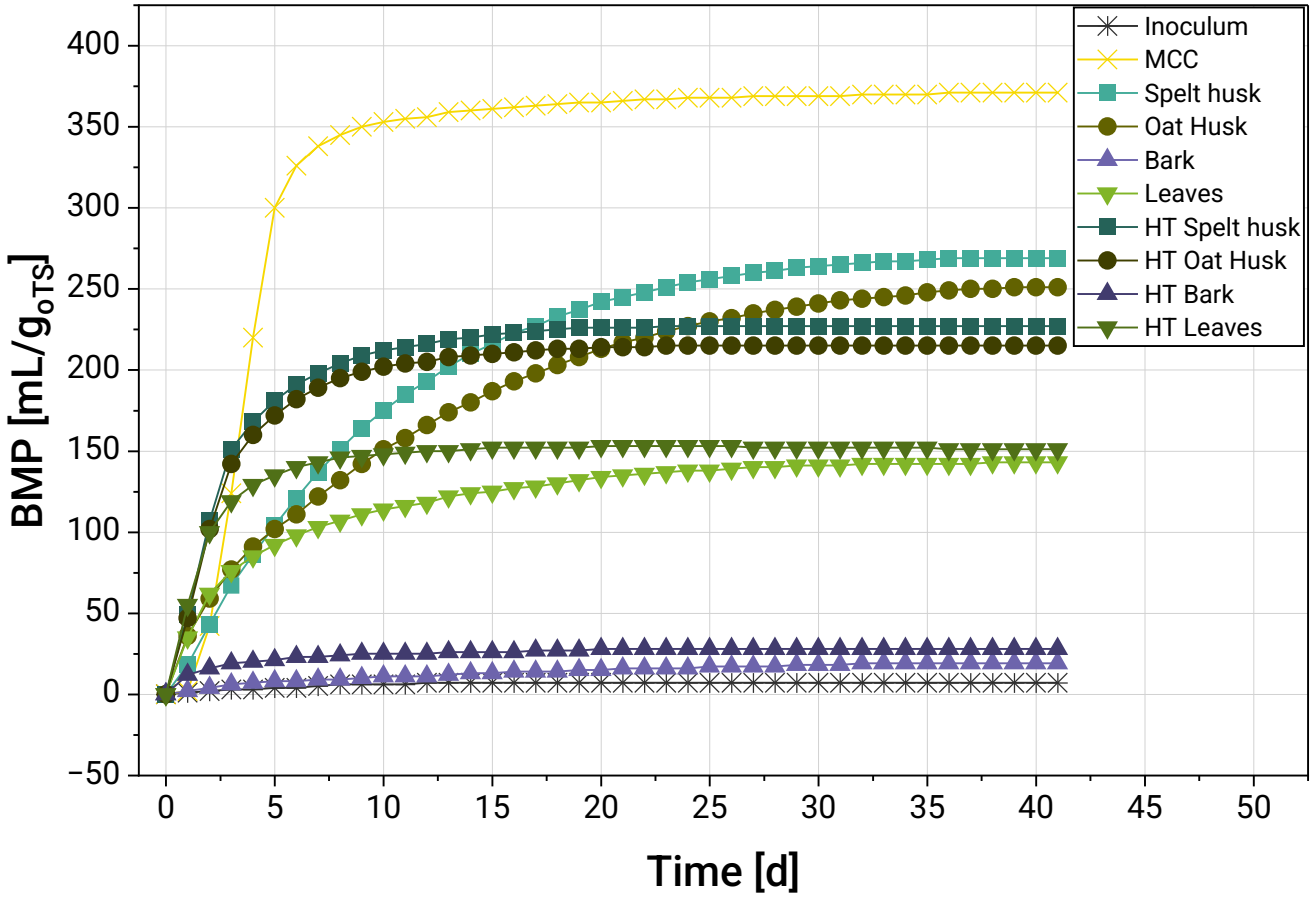
- BMP tests for pre-treatment with feedwater pH of 12
- Husks show similar performance, spelt husks slightly better (270 mL/g_{oTS})
- Bark forms almost no biogas (19 mL/g_{oTS})
- Leaves inbetween (143 mL/g_{oTS})



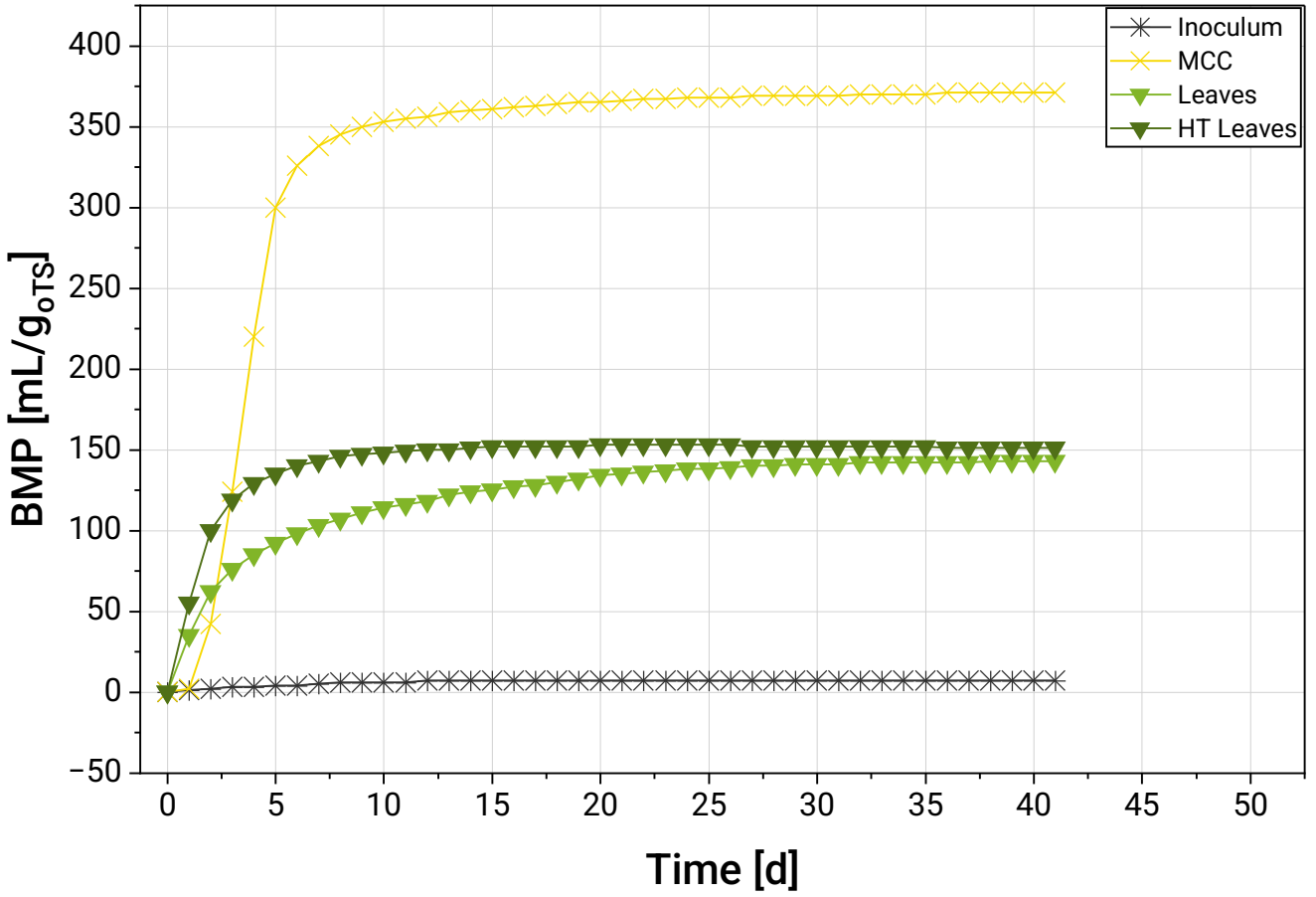
Influence of HTP

Biomass	BMP [ml/g_{oTS}]	BMP_{HT} [ml/g_{oTS}]	Δ
Spelt husk	269	227	-16 %
Oat husk	251	215	-14 %
Leaves	143	151	+6 %
Bark	19	28	+47 %

- In all cases: improved kinetics
  - ➢ Decomposition completed after 10-15 d
- ➢ Leaves selected for optimization.

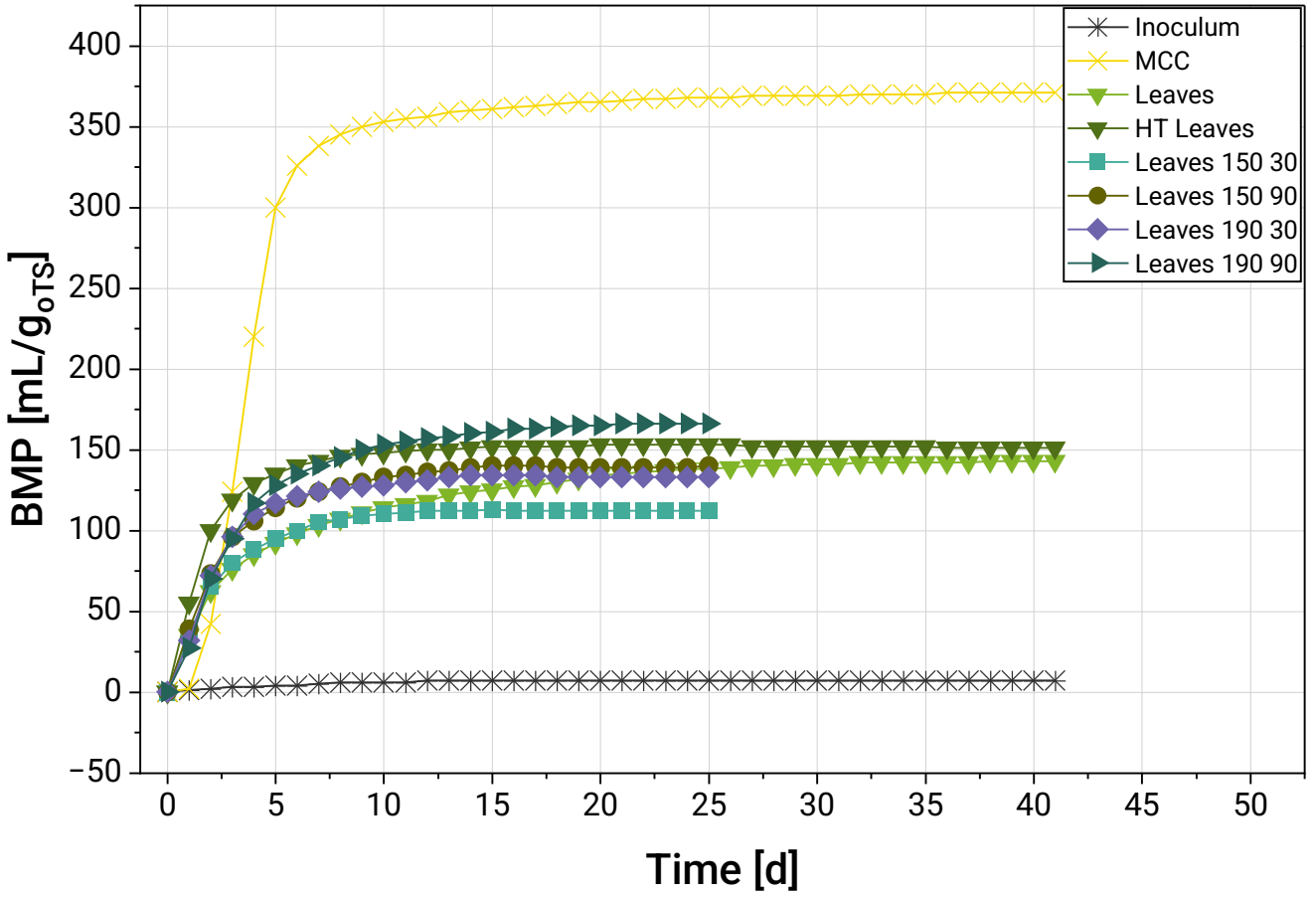


Optimization of pre-treatment

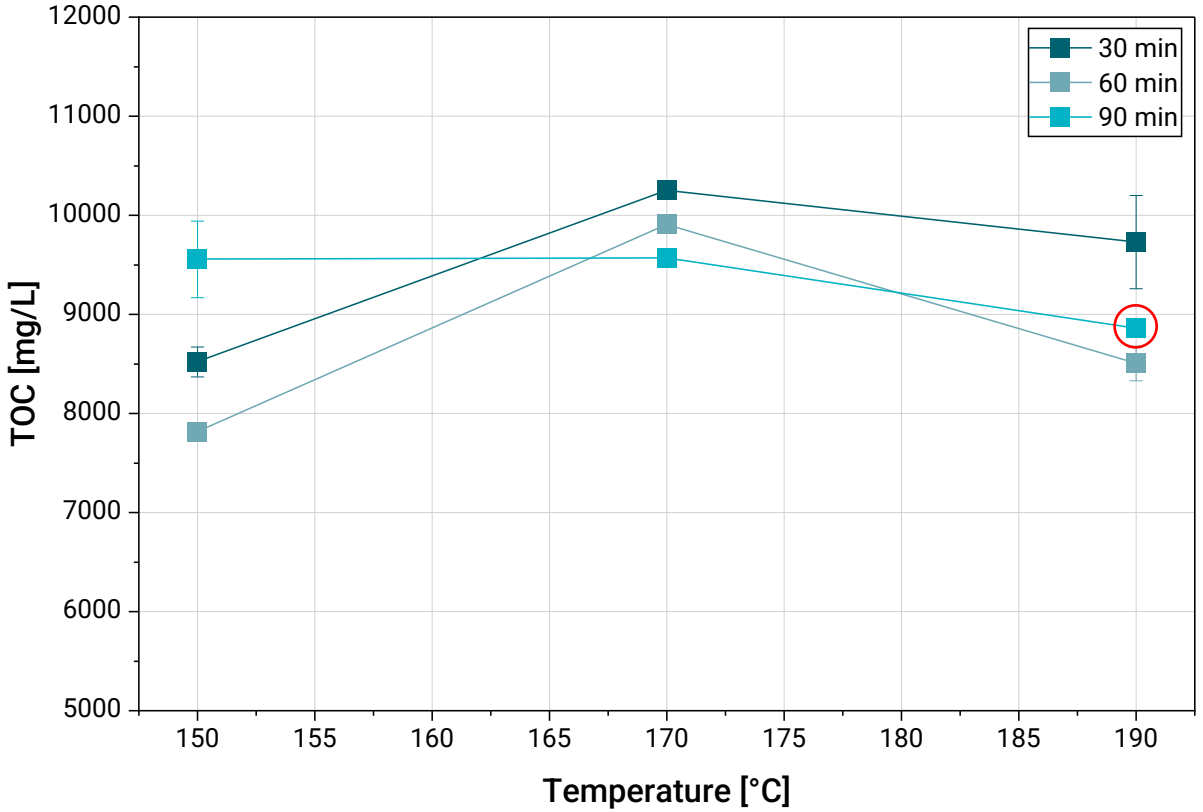


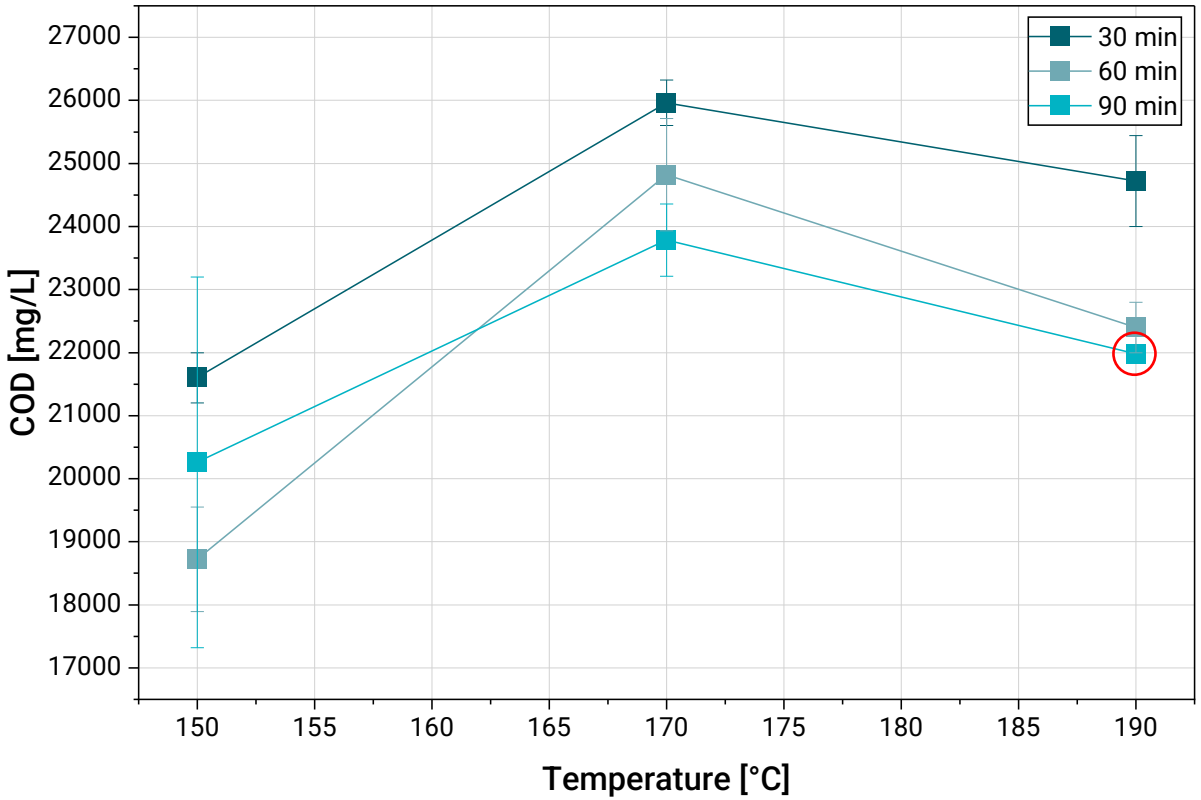
Optimization of pre-treatment

- BMP increases with severity
  - Highest increase (18 %) with a pre-treatment at 190 °C for 90 min
  - Similar relative uplift as found before with straw/manure



Optimization of pre-treatment





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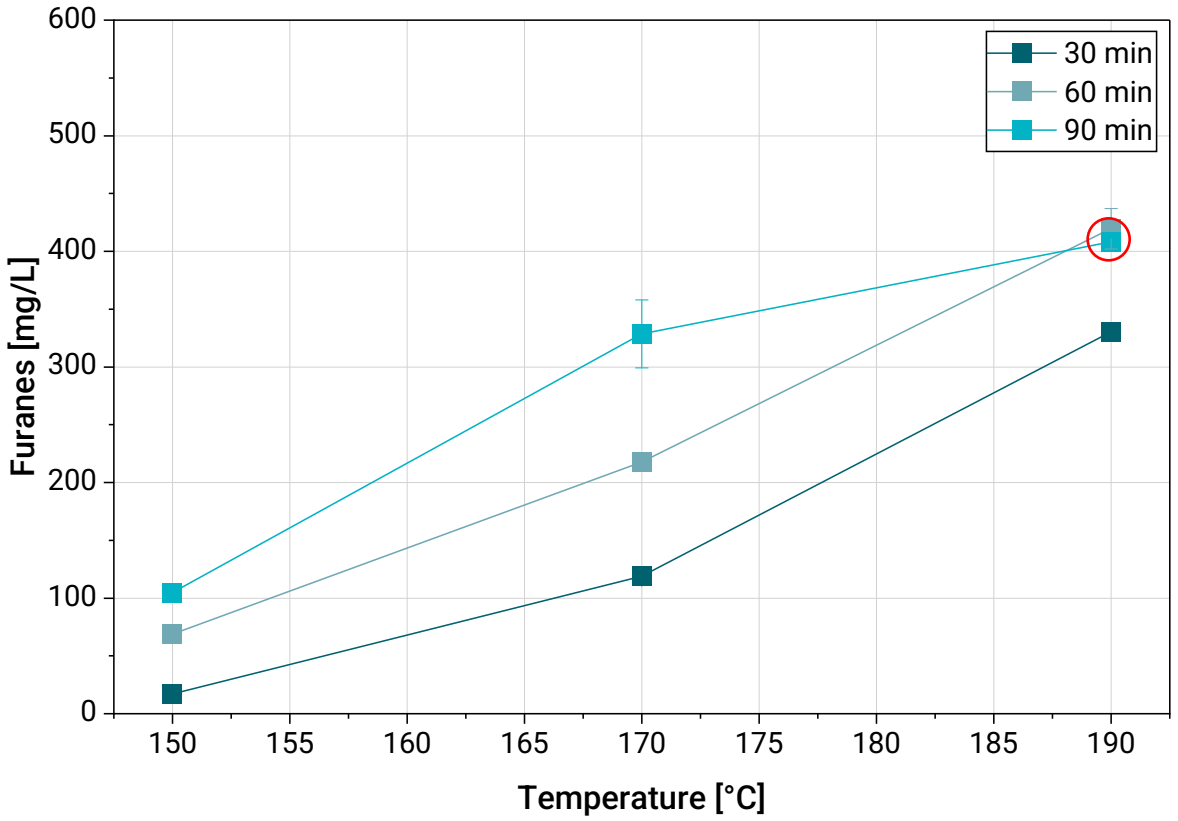
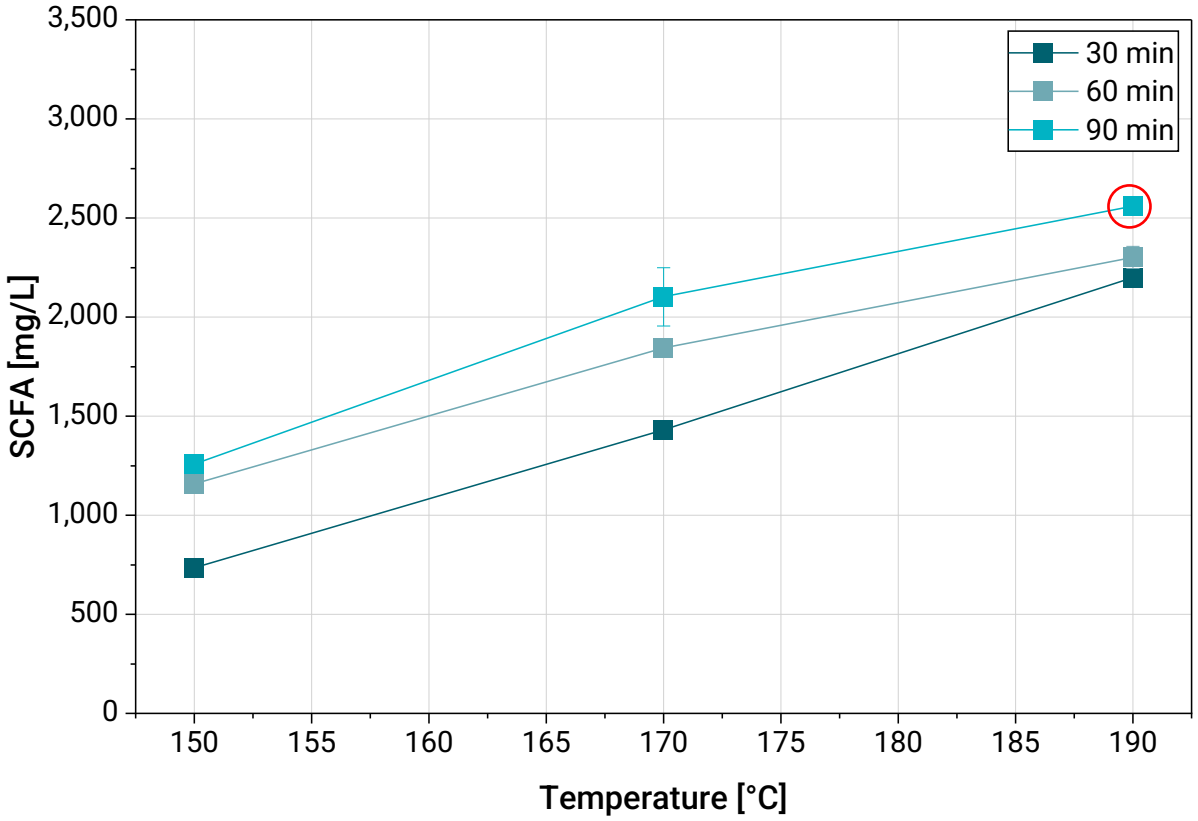
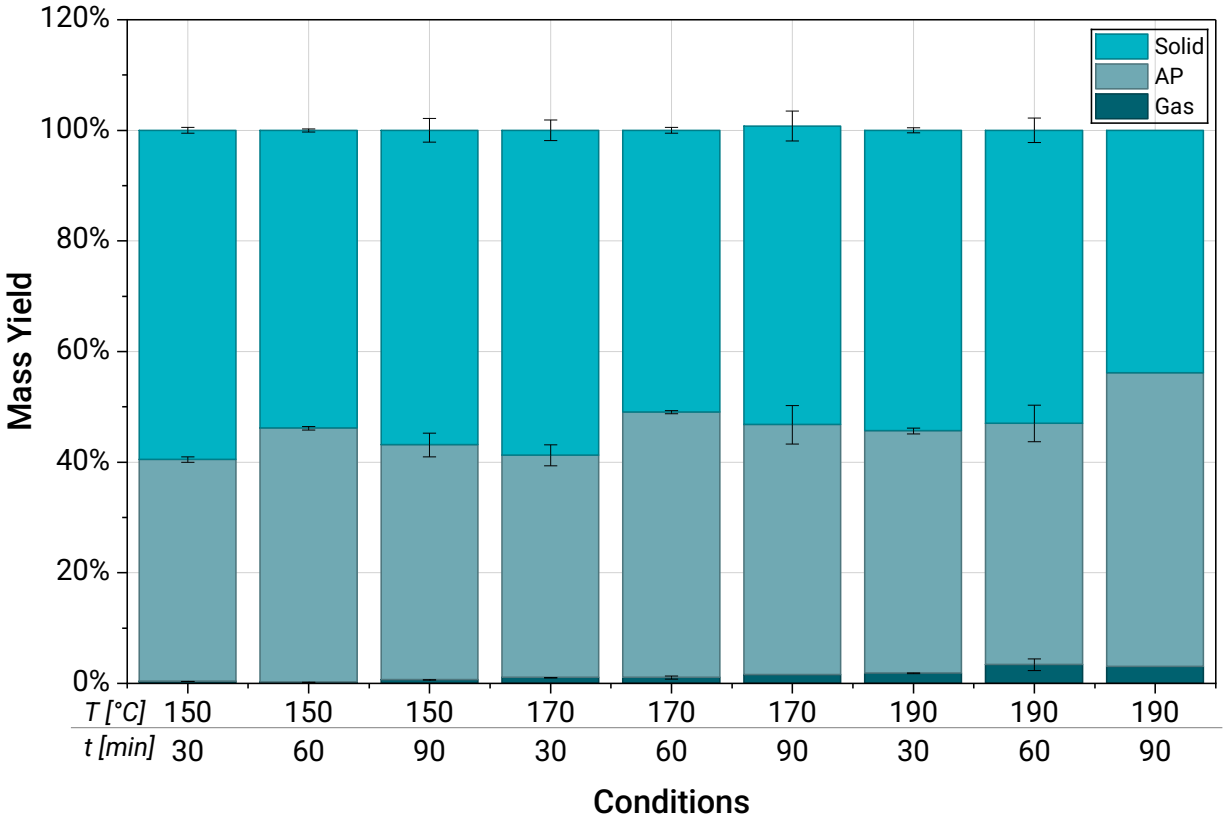
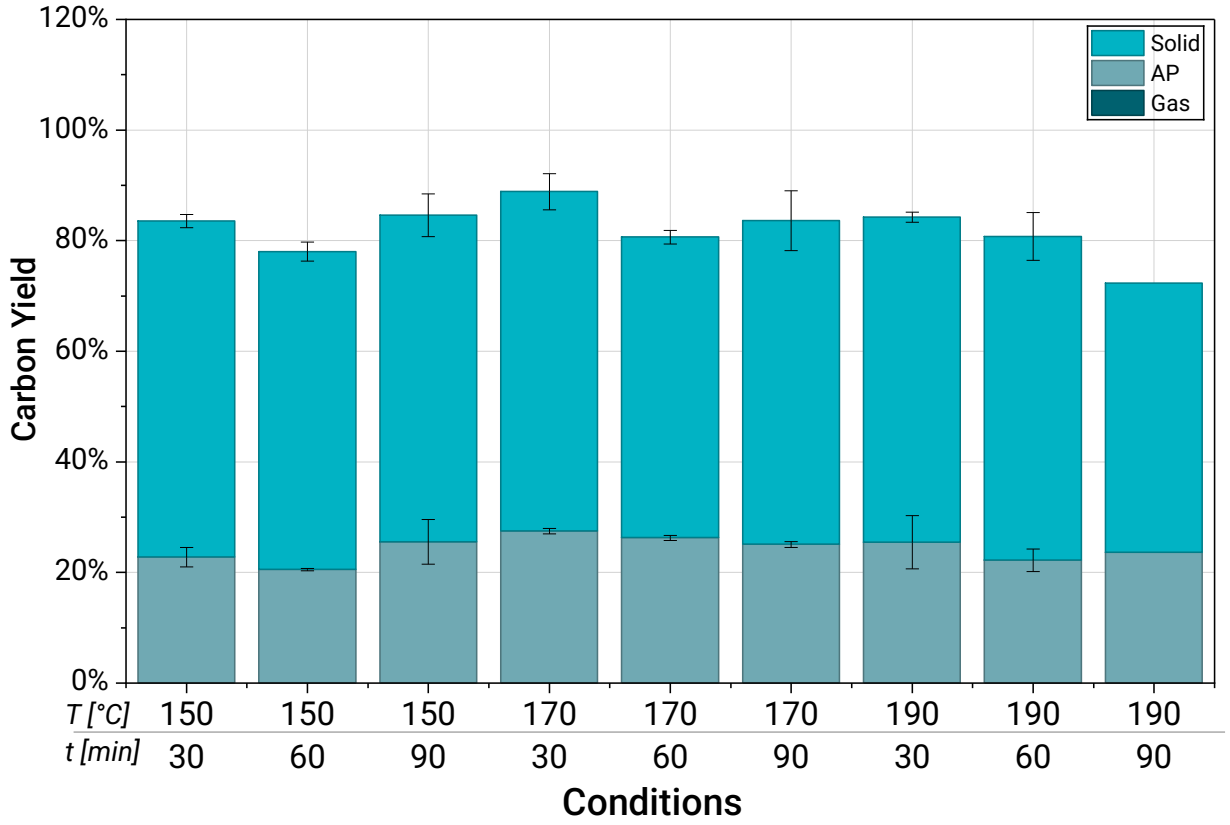


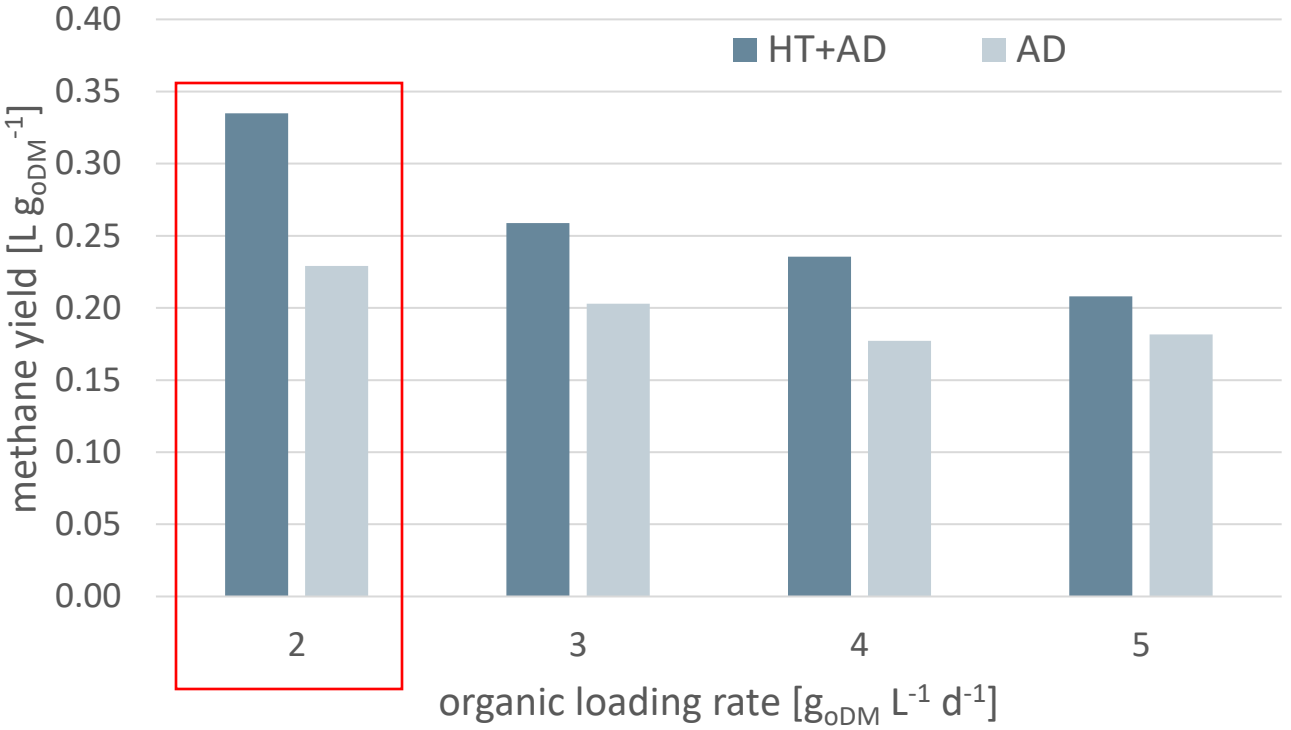


Optimization of pre-treatment





Pilot plant operation



Pre-treated material

- In pilot plant with straw/manure: up to 50 % more methane with pre-treatment!

*The results presented should be viewed with caution, as they are based on live plant data that is still being collected and analyzed. More comprehensive data and interpretations will be provided in future publications

Conclusion & Outlook

- BMP can be increased using hydrothermal pre-treatment by up to 18 %, depending on biomass and treatment conditions
- Simple derivations from TOC, COD, organic acid and 5-HMF concentration to BMP are difficult
- Findings in batch operation may underestimate continuous operation results

- Scale up of hydrothermal pre-treatment to 100 L scale
- Comparison of treated vs. untreated AD in continuous operation

Interested? Contact us!

More research on upscaling, economics, educt potentials, etc.

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