Deutsches Biomasseforschungszentrum

Biorefineries

Development of a purification cascade for industrial wood hydrolysates

Arne Gröngröft, Otto Laufer, Theresa Menzel | 21th edition of the International Conference on Renewable Resources & Biorefineries | Turku | 02. - 04.06.2025

Motivation

Wood hydrolysates

- Obtained by hydrolysis of hemicellulose, the second most abundant polysaccharide
- Especially innovative wood fractionation leads to interesting hemicellulose hydrolysates
- Rich in pentose mono- and oligosaccharides but also hexoses

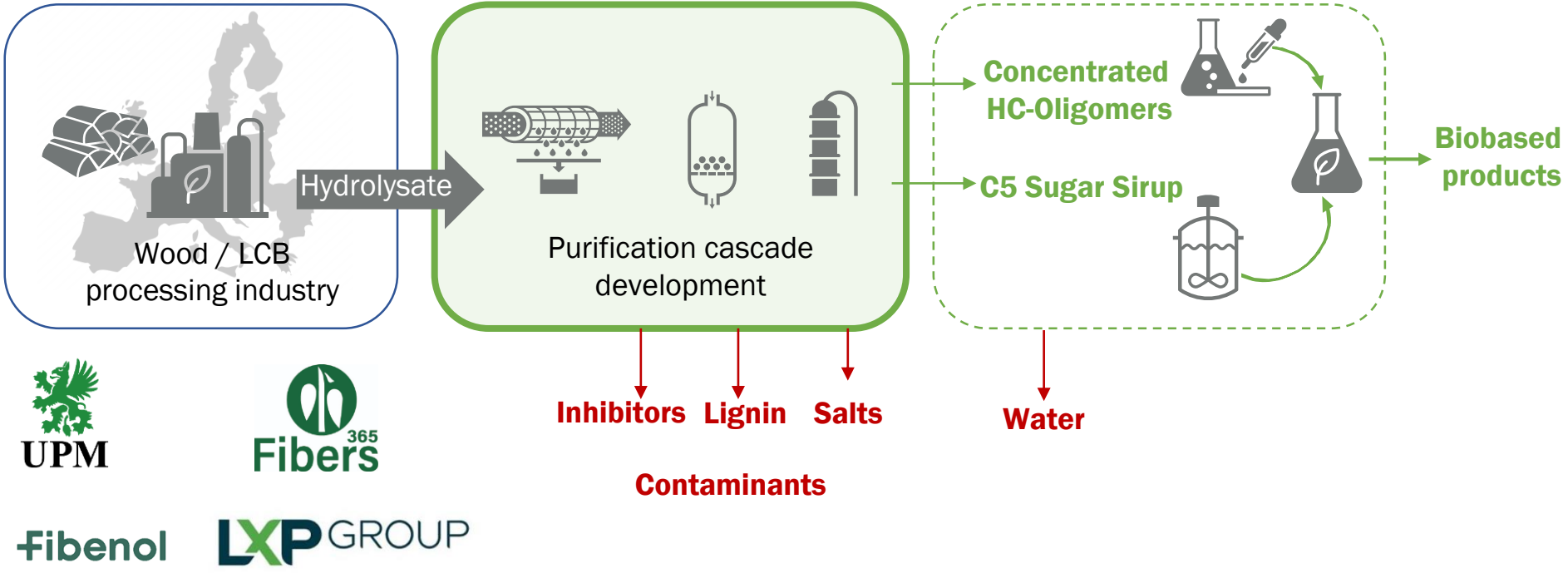
Commercial valorization faces several challenges

- Heterogeneous composition: depending on type wood and pretreatment technique
- Contain impurities like lignin, phenols, color, smell, salts
- Low concentration of sugars

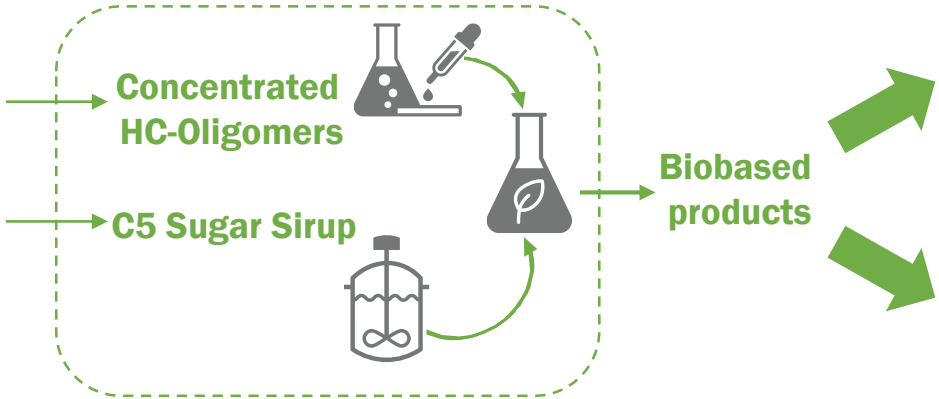
Chances

- Increase economic viability of biorefineries
- Provide large amounts of relatively low cost, sustainable feedstock for diverse applications

Aims


Wood / LCB
processing industry
Hydrolysate
Purification cascade
development
Concentrated
HC-Oligomers
C5 Sugar Sirup
Biobased
products
Inhibitors
Lignin
Salts
Water
Contaminants

Aims



NEXTSTEP

Biobased chemicals (aMVL, 3MdVL, 3MPD) for polymer applications

https://nextstep-cbe.com/

HEMIC-OAT

Biobased coatings, surfactants & buildings blocks

Wood adhesives

https://hemicoat.eu/

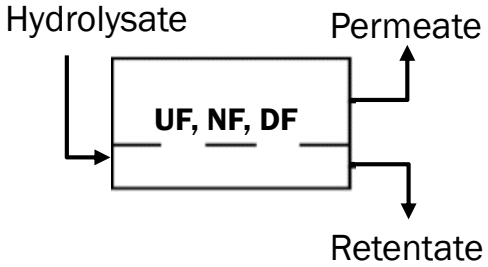
https://nextstep-cbe.com/ - HORIZON-JU-CBE-2023-IA-06
https://hemicoat.eu/ - Cornet Collective Research Network

Industrial hydrolysates from lignocellulosic biomass

Biomass	Hardwood & agricult. residues
Compounds	Range of concentration (in % dm)
C5 - Monosaccharides	16.1 – 55.1
C6 - Monosaccharides	14.0 – 30.5
Total Oligosaccharides	2.14 – 10.7
Carboxylic acids	0.6 – 2.2
Soluble lignin	6.5 – 15.3
Furfural	0.02 – 2.7
5-HMF	0.3 – 0.55
Anorganic acids	1,3 – 4.9

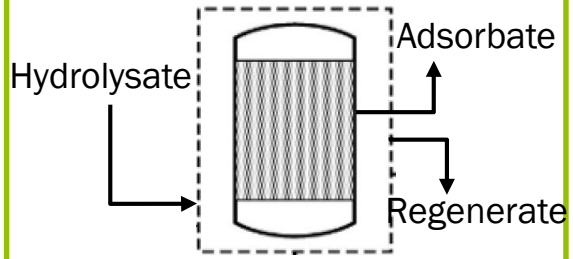
Purification steps for fermentation application of hydrolysates

Membrane Filtration



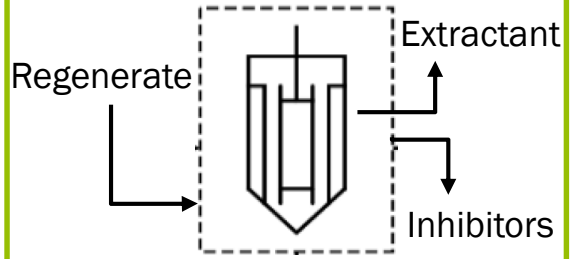
- Separation of macromolecules & particles
- Separation of oligo- and monosaccharides
- Sugar concentration

Adsorption



- Retention of Inhibitors: Lignin, Furans and Phenolic compounds
- Separation of carboxylic acids & ions

Distillation



- Sugar concentration
- Lignin recovery
- Ethanol recovery from adsorption

UF – Ultrafiltration, NF – Nanofiltration, DF - Diafitration

Adsorption, experimental setup

Adsorbent

- Specific surface area: 1100 m^2 g^{-1}
- Medium pore size
- EVB-DVB matrix
- 1 bed volume = 1 m^3 m^{-3} resin

Equipment

- Column: YMC-PilotPlus
- Bed height: 600 mm
- Bed volume: approx. 9 L
- Pump: Masterflex l/P

	Medium	Throughput (BV)	Flowrate (BV h^{-1})
Flushing	H_2O	2	5
Loading	C5 Hydrolysate	10	5
Flushing	H_2O	3	5
Regeneration	50wt% EtOH:H_2O	4	2
Flushing	H_2O	3	5

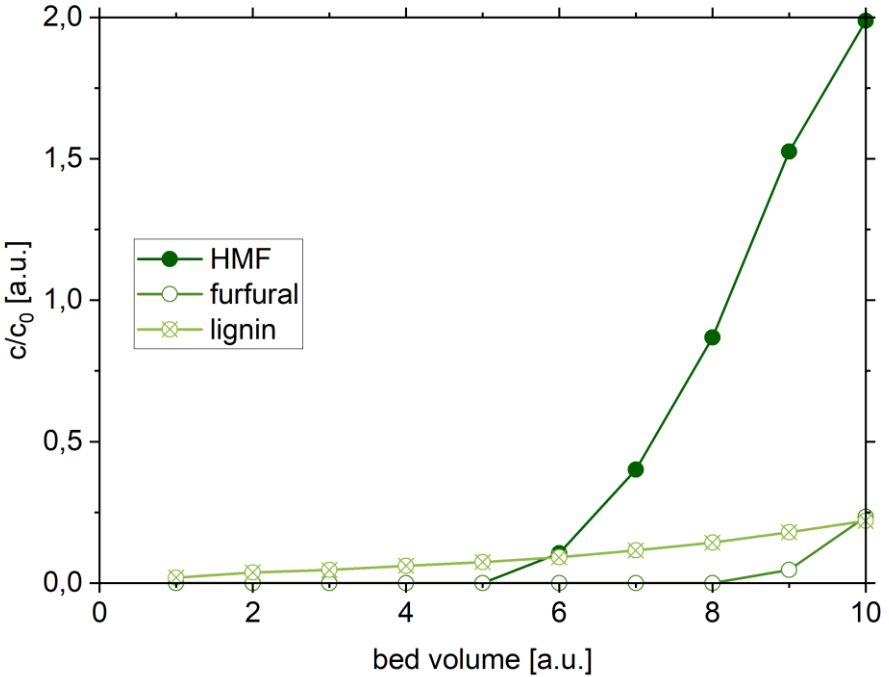
Adsorption, loading cycle

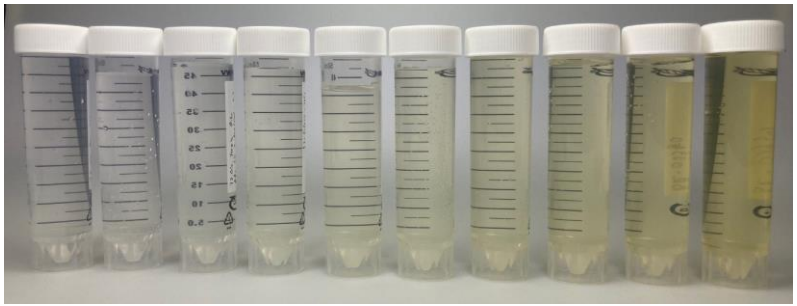
Aim: retention of phenolic compounds

Compound	Avrg. Retention after 10 BV in %
5-HMF	40
furfural	95
lignin	89
C5 monomers	0
C6 monomers	0

Conclusions:

- No sugars are retained over the whole cycle
- HMF breaks through around 6 BV loading
- Furfural breaks through around 9 BV loading
- Continous breakthrough of lignin





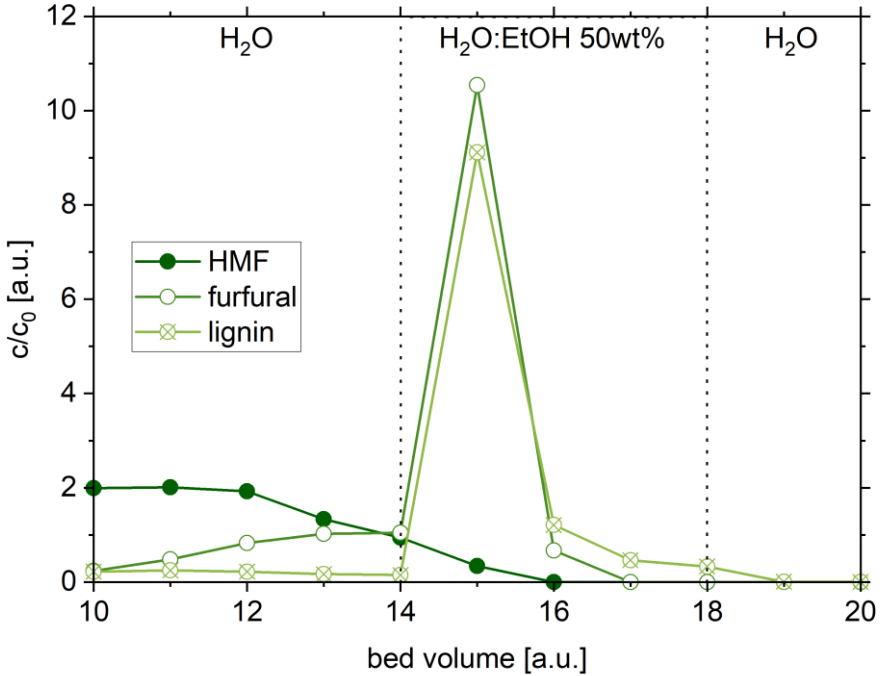
Adsorption, regeneration cycle

Aim: removal of retained compounds from resin

Inhibitor	Recovery
5-HMF	complete
furfural	complete
lignin	92 %

Conclusions:

- Lignin and furfural can be sufficiently removed by EtOH/H_2O
- HMF was mostly removed during flushing step with H_2O

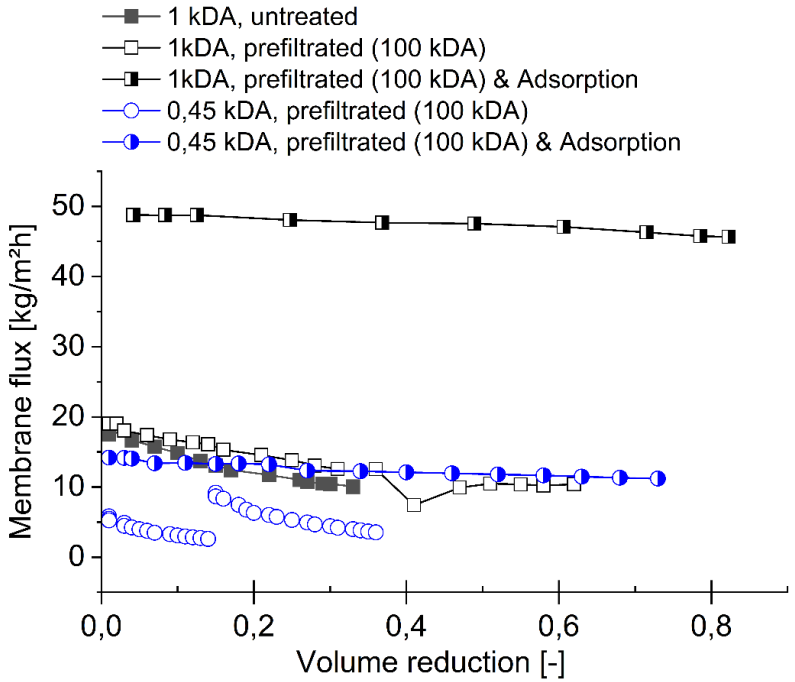


Membrane filtration, improving throughput in tight ultrafiltration

Aim: Separation of monosaccharides (~150 – 180 Da) from oligosaccharides (> 300 Da)

Tests on 2 ceramic membranes:

- 450 Da
- 1000 Da
- with different pretreatments



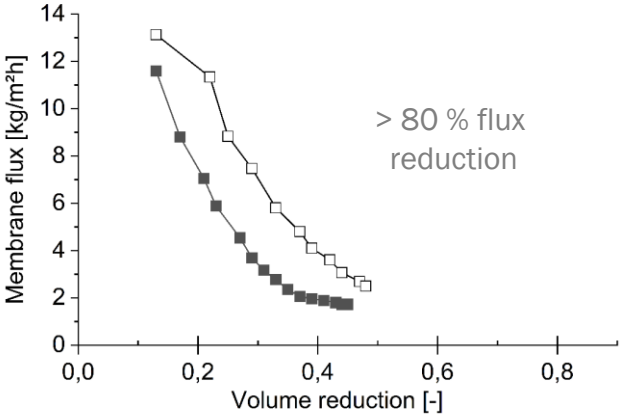
- Prior adsorption significantly reduces fouling
- Smaller pores → lower flux

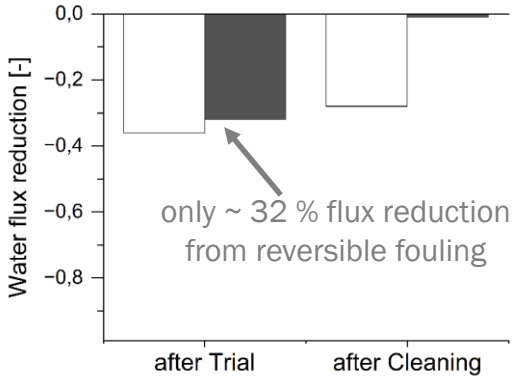
All experiments were conducted under the same operation conditions: Temperature: 40 °C / Transmembrane pressure: 4 bar / Cross flow velocity: 2.3 m/s
Flux reduction is related to initial measured flux within the experiment.

Membrane filtration, sugar concentration with nanofiltration

Aim: Concentration of monosaccharides (~150 – 180 Da)

- 2 repetition tests on spiral wound membrane (150 Da)
- Hydrolysate pretreated by adsorption

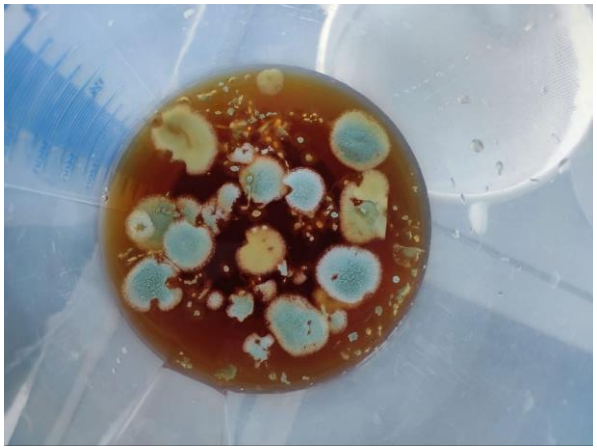




- Concentration by factor 2 → sign. flux reduction
- Irreversable fouling
- So far NF for concentration not suitable

Application tests

Fermentation tests



- Faster spoilage
- >800L purified for tests, results pending

Wood adhesive formulation

Lapshear tests with commercial xylan /chitosan mixtures:

Formulation	Average strength at break
Commercial wood adhesive	3.1 MPa
70 % xylane	2.13 MPa
1 % xylane – 2 % chitosan (1:3)	0.55 MPa
1 % xylane -0,5% NFChit (1:1)	0.1 MPa

- High potential with concentrated, long oligo-/polysaccharides

Tested at CELABOR, BE, www.celabor.be

Conclusions

Adsorption:

High selectivity: no sugars are retained, clear HMF and furfural break throughs, continous breakthrough of lignin

EtOH:H_2O suitable for desorption of lignin and furfural, HMF already removed with H_2O

Ultrafiltration:

Removal of lignin and phenolic compounds leads to significantly reduced fouling

Decision on suitable MWCO still open

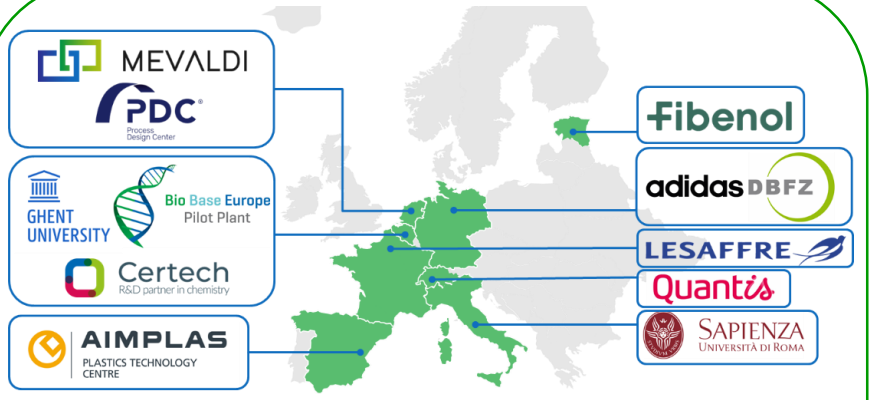
Outlook:

Fermentation and adhesives tests on purified samples ongoing

Removal of salts, organic acids

Partners and funding

NEXTSTEP



Circular Bio-based Europe Joint Undertaking

Bio-based Industries Consortium

Co-funded by the European Union

The NEXT-STEP project is supported by the Circular Bio-based Europe Joint Undertaking and its members, under GA 101157081

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Gesellschaft für Chemische Technik und Biotechnologie e.V.

celabor

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MATERIA NOVA Materials R&D Center

Gefördert durch:

Bundesministerium für Wirtschaft und Klimaschutz

aufgrund eines Beschlusses des Deutschen Bundestages

IGF
INDUSTRIELLE GEMEINSCHAFTSFORSCHUNG

Wallonie

SPW
Service public de Wallonie

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Collective Research Networking

Funding code: 01IF00399C 2310168

Thanks to the team, the partners and for the funding!