Progress task 2.4

Wouter Huijgen (ECN)

MacroFuels Progress Meeting, Wageningen
10th Jan, 2017

Task 2.4: Fractionation & chemical treatment

- **Goal: Liquefaction of seaweed species by hydrolysis, to produce sugar syrups for ABE fermentation (WP3) and pre-cursors for furanic based fuels (WP4)**

- Task 2.4.1 (ECN, AVT) **Liquefaction** of seaweed by **light (organic) acid or base hydrolysis** with optimization of temperature, time, catalyst and biomass loading. For fermentative purposes, the carbohydrate molecules need to be hydrolysed to their monomers. Using **organic acids** which are needed for fermentation, such as acetic acid, will be used to **produce sugar syrups suitable for fermentation**.

- Task 2.4.2 (AVT, DLO) **Mineral acid hydrolysis** of seaweed for liquefaction and partial hydrolysis will be tested at various temperature, time, acid and biomass loading. In some cases, organic acids are inhibitors for fermentation or thermochemical conversions.

→ **T 2.4.1 + T2.4.2 (ECN, AVT, DLO)** Liquefaction of seaweed by hydrolysis, to produce sugar syrups and pre-cursors for furanic based fuels

Tasks

MACROFUELS

- Task 2.4.3 (ECN, MATIS OHF, DLO, AVT) Based on results from the tasks above**, combinations of chemical and enzymatic treatments** will also be explored as well as sequential mild aqueous extractions **combined with mechanical treatment such as shredding, reactive extrusion etc.** for full hydrolysis of seaweed to monomeric sugars and processable oligomeric sugar streams.

- Task 2.4.4 (DLO) **mechanical treatment** to yield sugar solutions for WP 3 and 4 will be **optimized** using among **others screw presses, stirred reactors, extruders and similar equipment** to yield solutions which contain sugars that are suitable for fermentation and or thermochemical conversions.

Contribution ECN

- Goal task 2.4 : production of sugar syrups for fermentation (WP3) and pre-cursors for furanic based fuels (WP4)
- Focus ECN 2016: tasks 2.4.1 & 2.4.2: liquefaction of seaweed by hydrolysis to produce monomeric sugars.
- Focus ECN 2016: mineral acid-catalysed thermochemical saccharification (starting point for MacroFuels based upon experience from previous projects, later focus on alkaline liquefaction and combined chemical-enzymatic saccharification).

Fractionation Saccharina

- Tests performed with fresh seaweed (1:1 w/w)
  - Water extraction, 80C, 1hr (extraction of mannitol and laminarin) (conditions*).
  - Saccharification: 120 °C, 0.1M H_2SO_4, 0.5hr (20L reactor).
  - Alkaline liquefaction: 80 °C, Na_2CO_3, 2.5hr.

- Product liquors delivered to WUR-FBR for ABE fermentation (WP3). However, concentration of sugars turned out to be too low due to inferior quality seaweed (high ash, low carbohydrates).

** J. van Hal & W.J.J. Huijgen (2012), Process for mannitol extraction from seaweed, patent NL 2009482*

Saccharification Palmaria

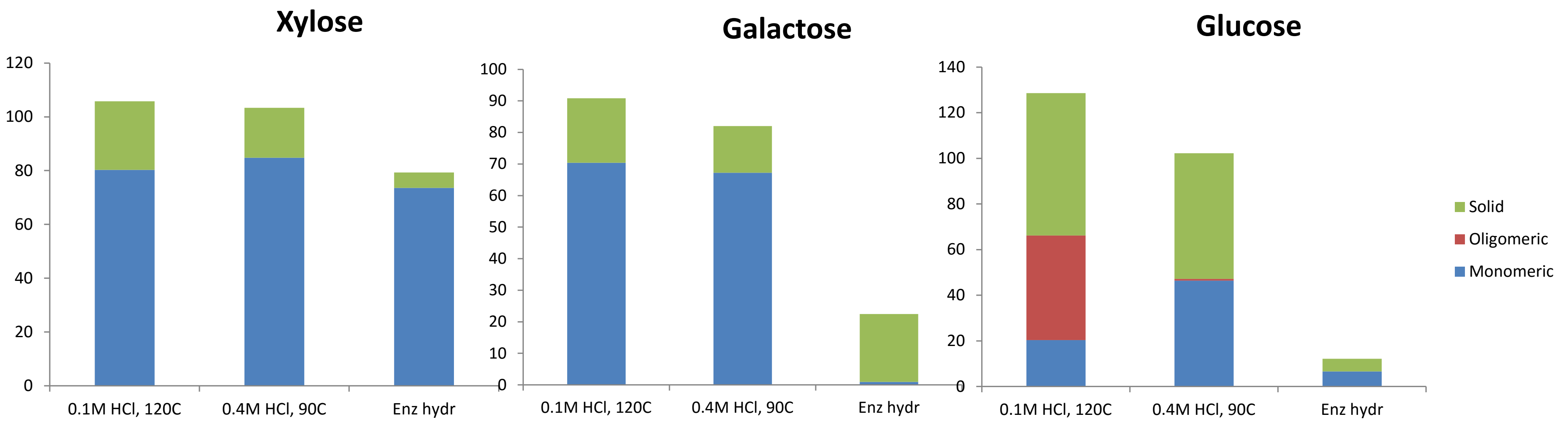
- Hydrolysis tests performed with fresh seaweed (1:1 w/w):
  1. 120 °C, 0.1M HCl, 2hr (optimum previous project*).
  2. 90 °C, 0.4M HCl, 3hr (optimum literature)
  3. Enzymatic hydrolysis (Accellerase TRIO/XY), 24hr.

- Product liquors delivered to:
  - WUR-FBR, ABE fermentation (WP3): 1 & 3.
  - ECN, furfural conversion (WP4): 1 & 2.

** W.J.J. Huijgen, A. Lopez Contreras, J. van Doorn & J.W. van Hal (2017) Xylose production and fermentation from red seaweed (Palmaria palmata) (in preparation).*

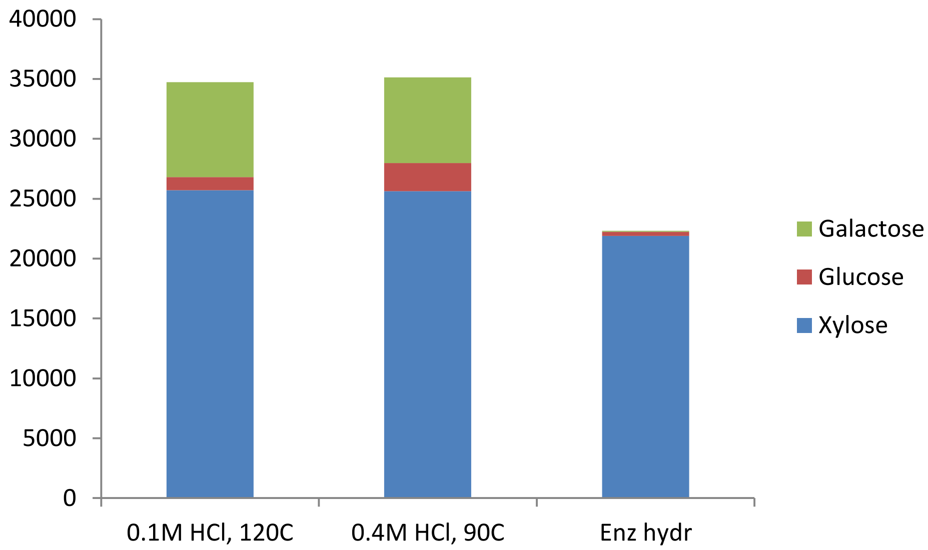
Yields & Mass Balances

- Residual solid: 33-36 dw%.
- Mass balances adequately closed for HCl-catalyzed processes, except Glc.
- Yield monomeric Xyl up to 85% and Gal up to 70%.
- Both HCl-catalyzed processes perform equally for Xyl and Gal.
- Xylan-backbone *P palmata* hydrolysable with commercial enzymes. However, no hydrolysis activity for floridoside and glucobiose (no cellulose?).



Composition Product Liquors

- Up to 35 g/kg monomeric carbohydrates in product liquors.
- In addition, dimers and oligomers.
- No analysis performed for salts etc.
- Target of 60 g/L sugars achievable (by reduction process liquor and purification/concentration).



Saccharification Ulva

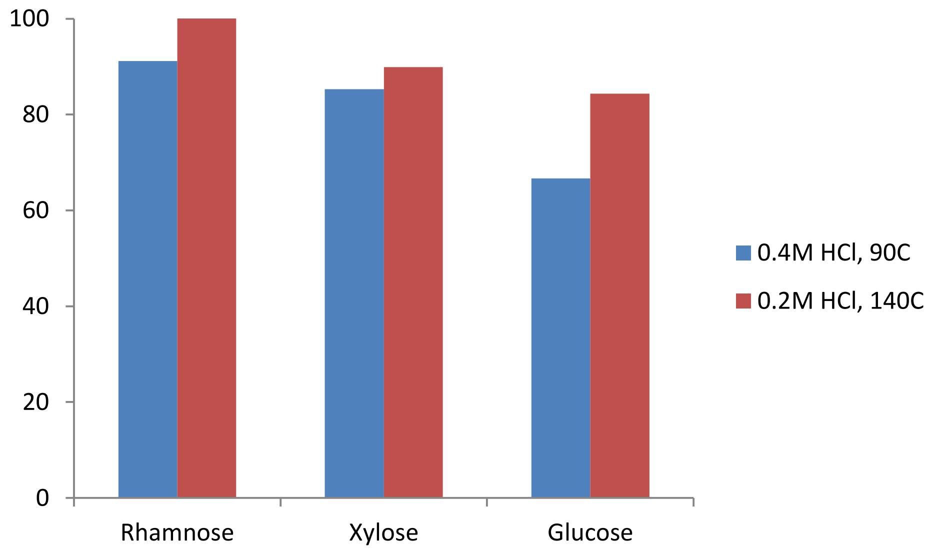
- Hydrolysis tests performed with fresh seaweed (1:1 w/w):
  1. 140 °C, 0.2M HCl, 1hr (optimum previous project*).
  2. 90 °C, 0.4M HCl, 3hr (optimum literature)

- Product liquors delivered to:
  - ECN, methylfurfural conversion (WP4): 1 & 2.

**) F. Groenendijk (Eds) with contributions from W.J.J. Huijgen, J.W. Dijkstra & J.W. van Hal (2016) North-Sea-Weed-Chain, Sustainable seaweed from the North Sea; an exploration of the value chain, report IMARES C055/16*

Yields & Mass Balances

- Monomeric yields of major carbohydrates (Glc, Rham, Xyl) of at least 85% possible.
- However, sugar concentrations in product liquors Ulva substantially lower (~5 g/kg) due to low carbohydrate content seaweed (in addition to high ash and sulphate content).



Planned activities 2017

- M2.2 (M24, Efficient hydrolysis of algal sugar polymers). Goal: Hydrolysis efficiency of 85% of all fermentable sugars.
- Effect of feedstock storage method on biorefining (testing frozen and air-dried seaweeds).
- Alkaline liquefaction and combined chemical-enzymatic saccharification of Kelps.
- Construction and testing of new seaweed processing line (incl upscaling from 20L (current volume) to 100L).

Seaweed Processing Line

Contained / ventilated

1. Cooled storage of feeds, intermediates and products
2. Cutting / milling/ washing
3. Biorefinery (100 Ltr autoclave)
4. Filtration and/or centrifugation
5. Concentration & purification (UF, rotavap)
6. Optionally precipitation (spray dryer , rotavap)

- Production facility for fractions (intermediates) from seaweeds for application tests:
  - Use of processes developed on smaller scale.
  - Modular / flexible unit.
  - From brown, red and green seaweeds to monomeric sugars (later expansion to co-products).
- ECN investment, but facility also to be used for large-scale production tests within MacroFuels (2018-2019).
- Planning: design and construction (Q1-2 2017), testing and adaptation (Q3-4 2017).

Progress task partners

- Jelle
- ….

Acknowledgement

MACROFUELS

Thank you for your attention!

This presentation is part of the MacroFuels project. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 654010

macrofuels@dti.dk