



**LNEG**  
Laboratório Nacional de Energia e Geologia, I. P.



HR EXCELLENCE IN RESEARCH



# Current status on advanced lignocellulose conversion to bioethanol and higher alcohols as biofuels

**Francisco Gírio**  
*Principal Researcher*  
*Head of Bioenergy Unit*  
*LNEG, Portugal*



**LNEG location:  
Lisboa, Portugal**

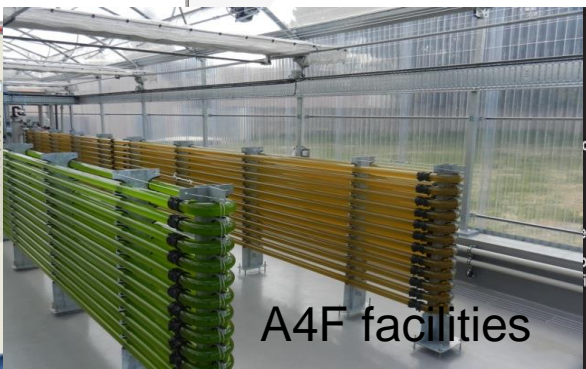
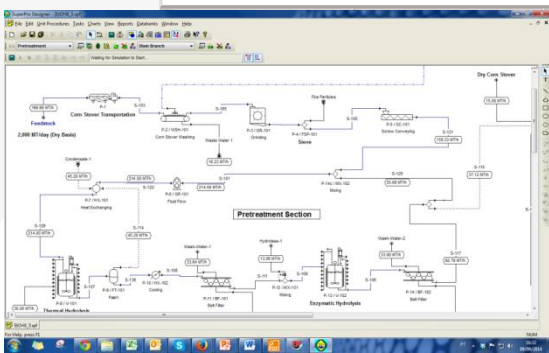
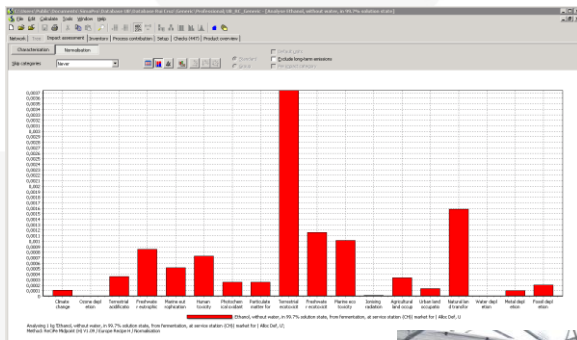
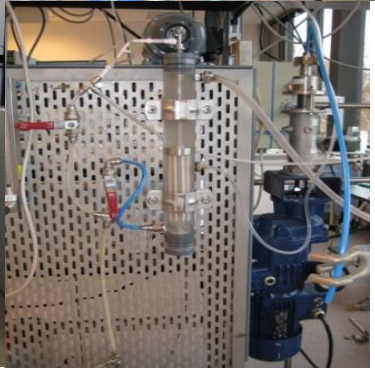
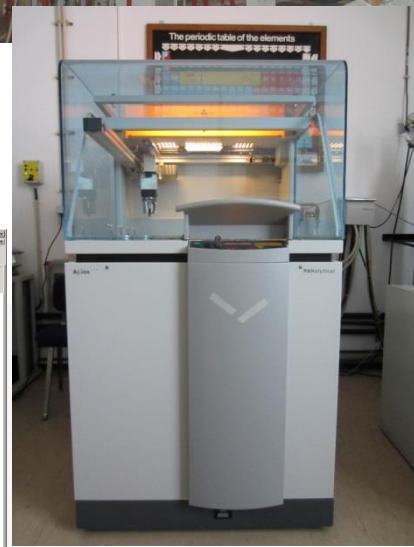


# UNIT OF BIOENERGY (LNEG)

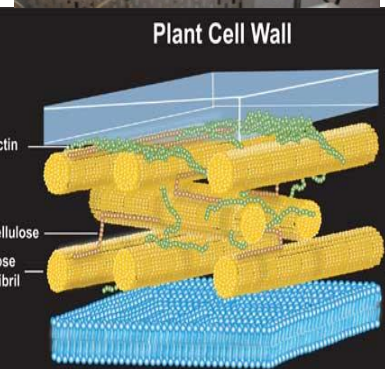
## Four Main Biomass R&D Areas:

1. Biomass availability & supply
2. Biofuels & bioproducts
3. Cell factories & enzymes
5. Bioenergy sustainability

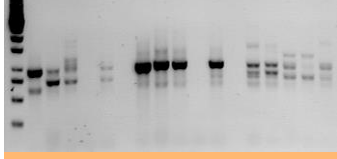
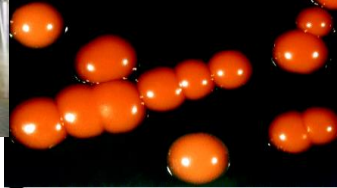
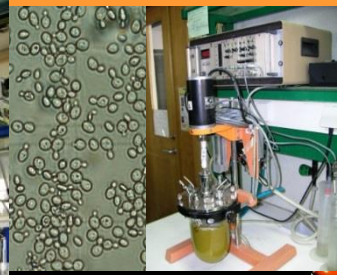
Staff= 30 researchers + 35 grantees and students.



A4F facilities



## Cell Factories & Enzymes

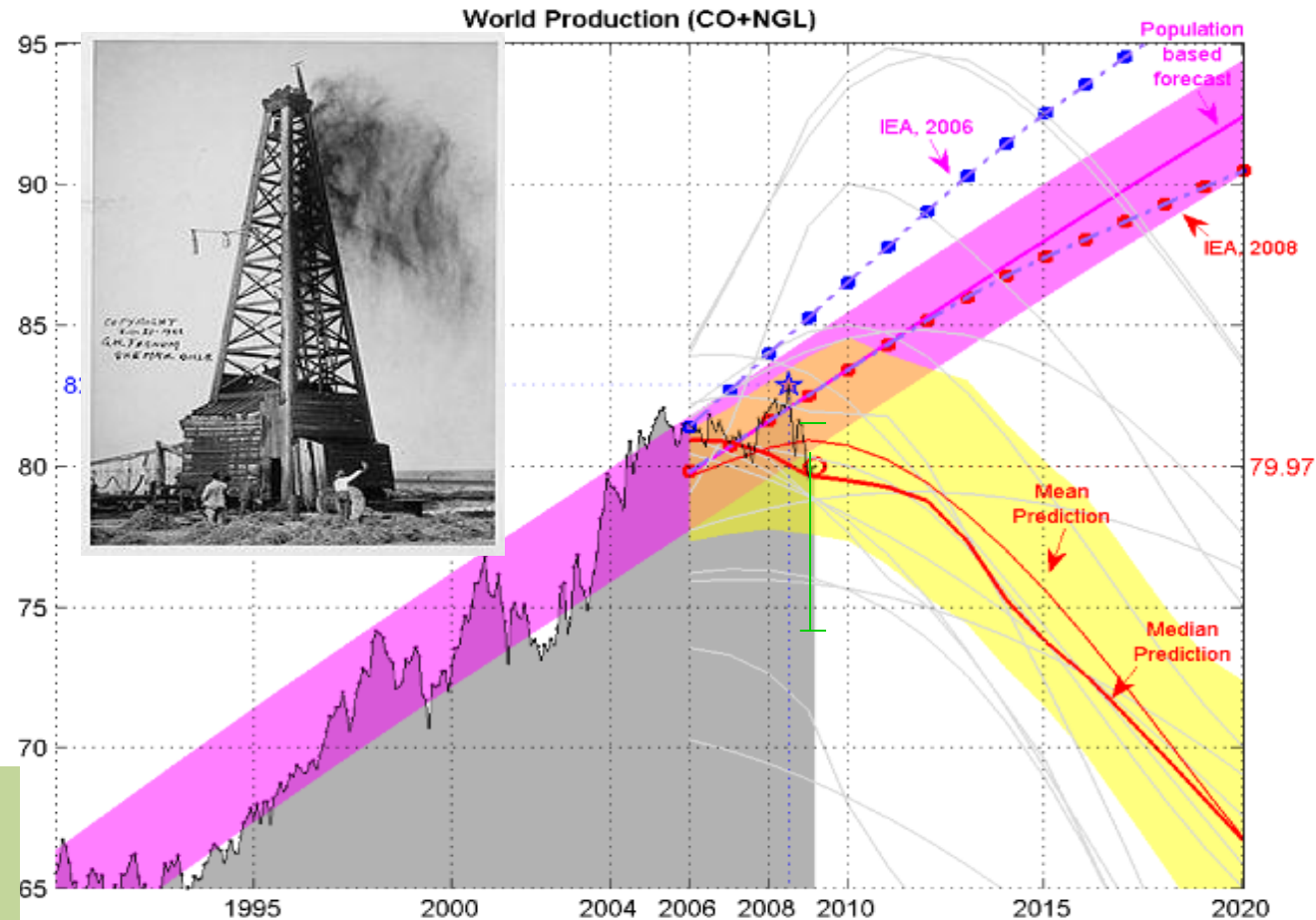


## Microbiology, Microbial Physiology & Molecular Biology



# Did crude oil reached a production peak?

23.10.2003



**World oil production (EIA Monthly) for crude oil + NGL. The median forecast is calculated from 15 models that are predicting a peak before 2020 (Bakhtiari, Smith, Staniford, Loglets, Shock model, GBM, ASPO-[70,58,45], Robelius Low/High, HSM, Duncan&Youngquist). 95% of the predictions sees a production peak between 2008 and 2010 at 77.5 - 85.0 mbpd (The 95% forecast variability area in yellow is computed using a bootstrap technique). The magenta area is the 95% confidence interval for the population-based model.**

**Break News: Saudi Arabia sees End of Oil Age coming and opens valves on the carbon bubble**

January 22, 2015 ("EnergyPost")

**However,  
This is not the point...!**

# The point is (I)....

Figure 1. World marketed energy consumption

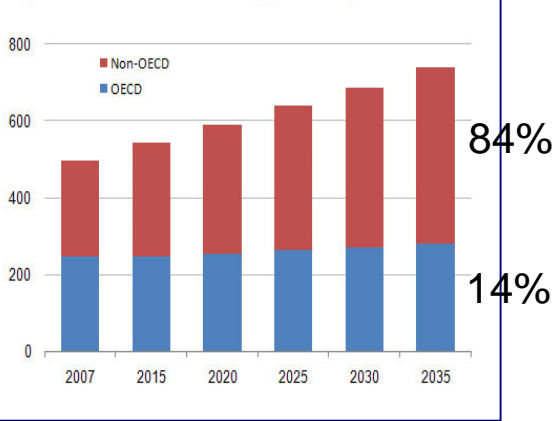
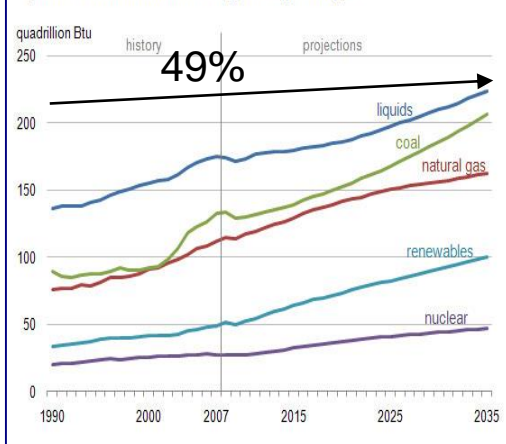


Figure 2. World marketed energy use by fuel type



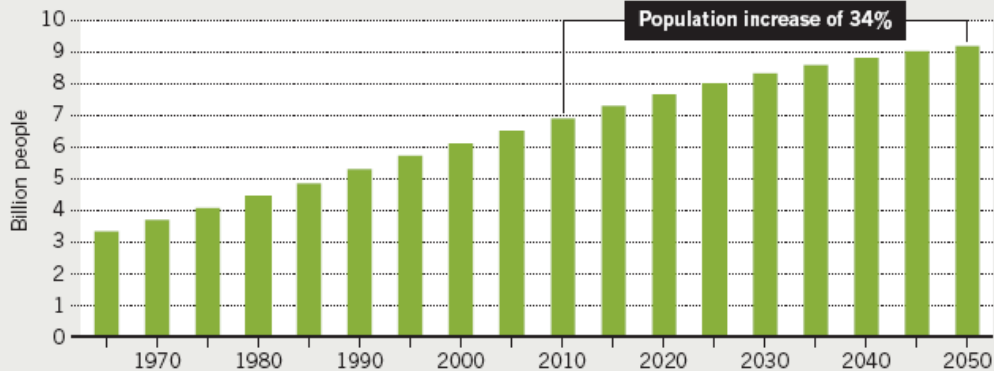
**World Energy demand will increase 49% before 2035...and much more before 2050 (1990 comparison)**

<http://www.eia.gov/oiaf/ieo/highlights.html>

## GROWING CHALLENGES

Between now and 2050 the world's population is predicted to increase to more than 9 billion people. Each person will also be consuming more calories per day and using more energy to power their lives. Food and fuel supplies will need to massively increase to meet these needs.

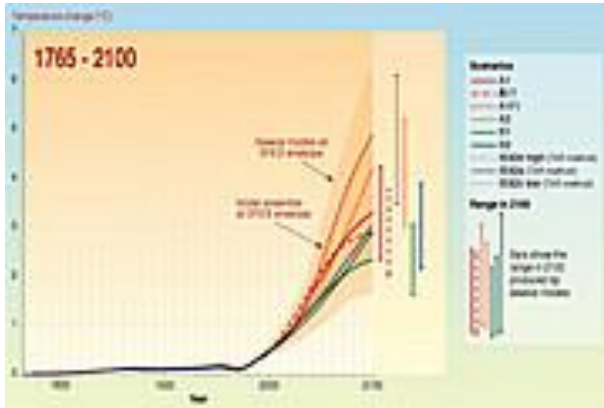
WORLD POPULATION



**World Population will increase 34% before 2050 (2010 comparison)**

# The point is (II)....

## global warming



*“The most powerful tool that the developed World has to reduce the CO2 emissions in the transportation sector is called Biofuels“*

**José Socrates**, Portuguese former prime-minister speaking as President of the European Union, Brussels, (In: *Diário Económico*, July 6th, 2007)



**Flooded village**



**Bleached coral**  
(high water temperatures)

# Environmental impact of the biofuel n generations

## LCA well-to-wheel (not considering LUC and ILUC)

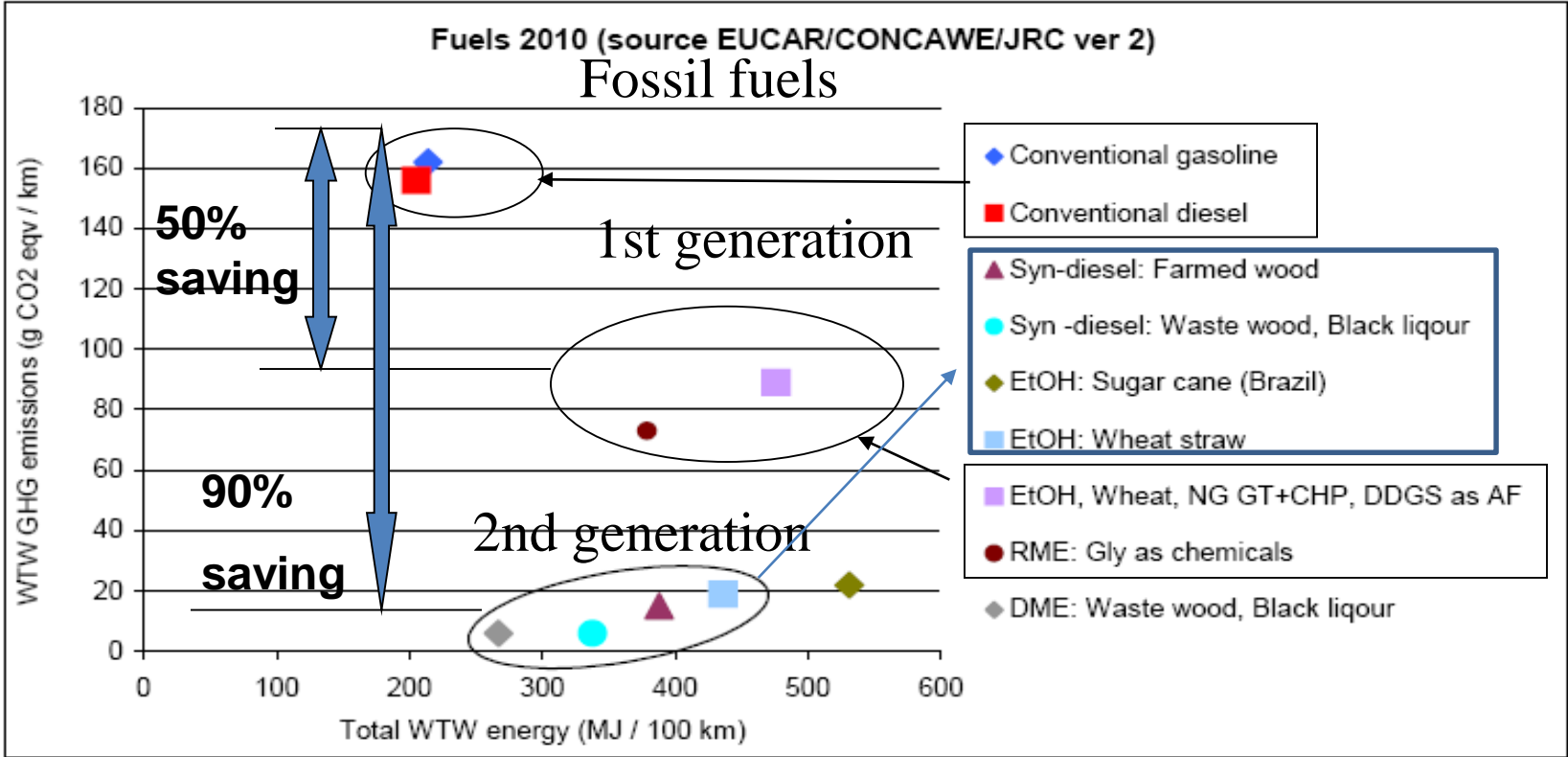
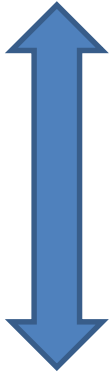


Figure 1.5. Well-to-wheel greenhouse gas emissions (in CO<sub>2</sub>-equivalents/km) versus total energy use for running a mid-size car over a distance of 100 km.

# Lignocellulosic-based Biofuel Biorefineries: What are the challenges for next 5-10 yrs?

Feedstock

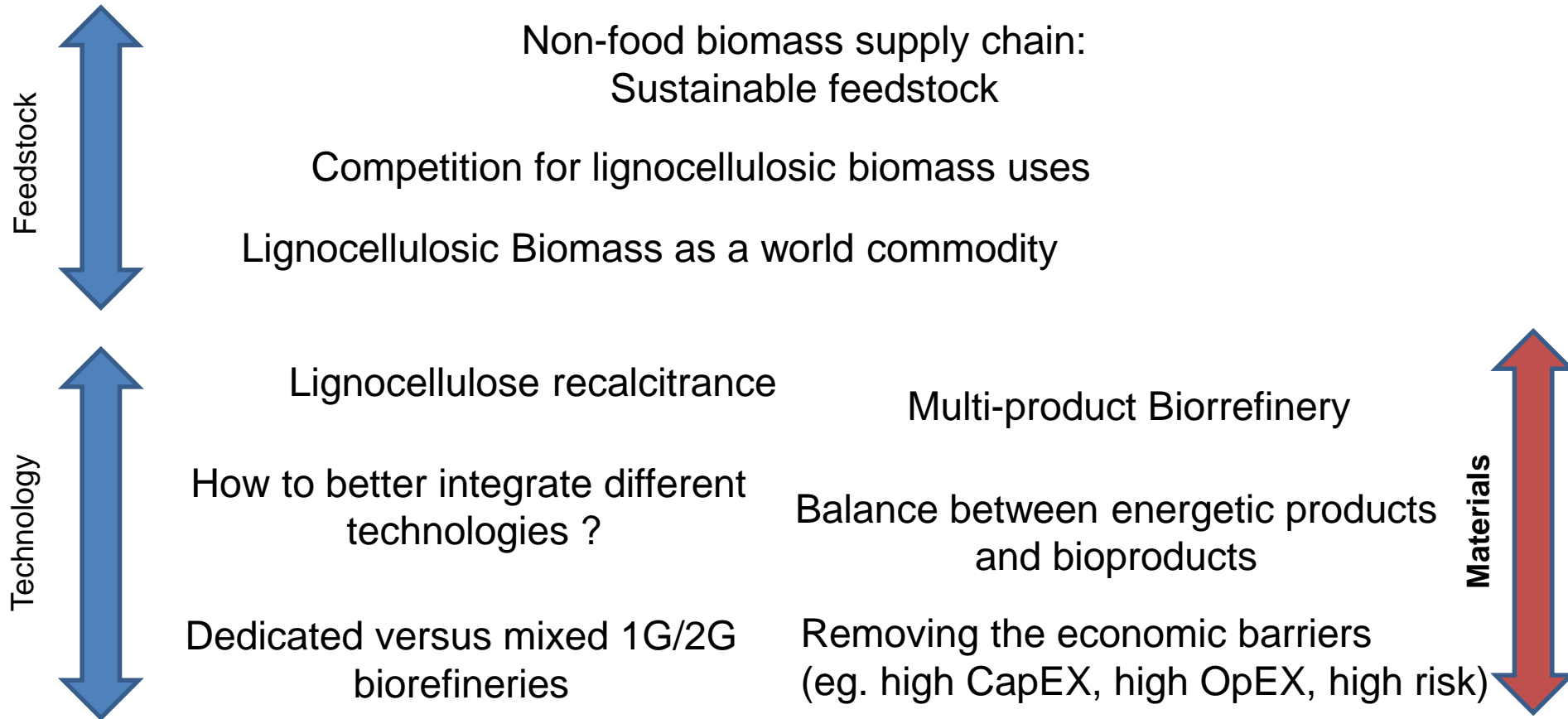


Non-food biomass supply chain:  
Sustainable feedstock

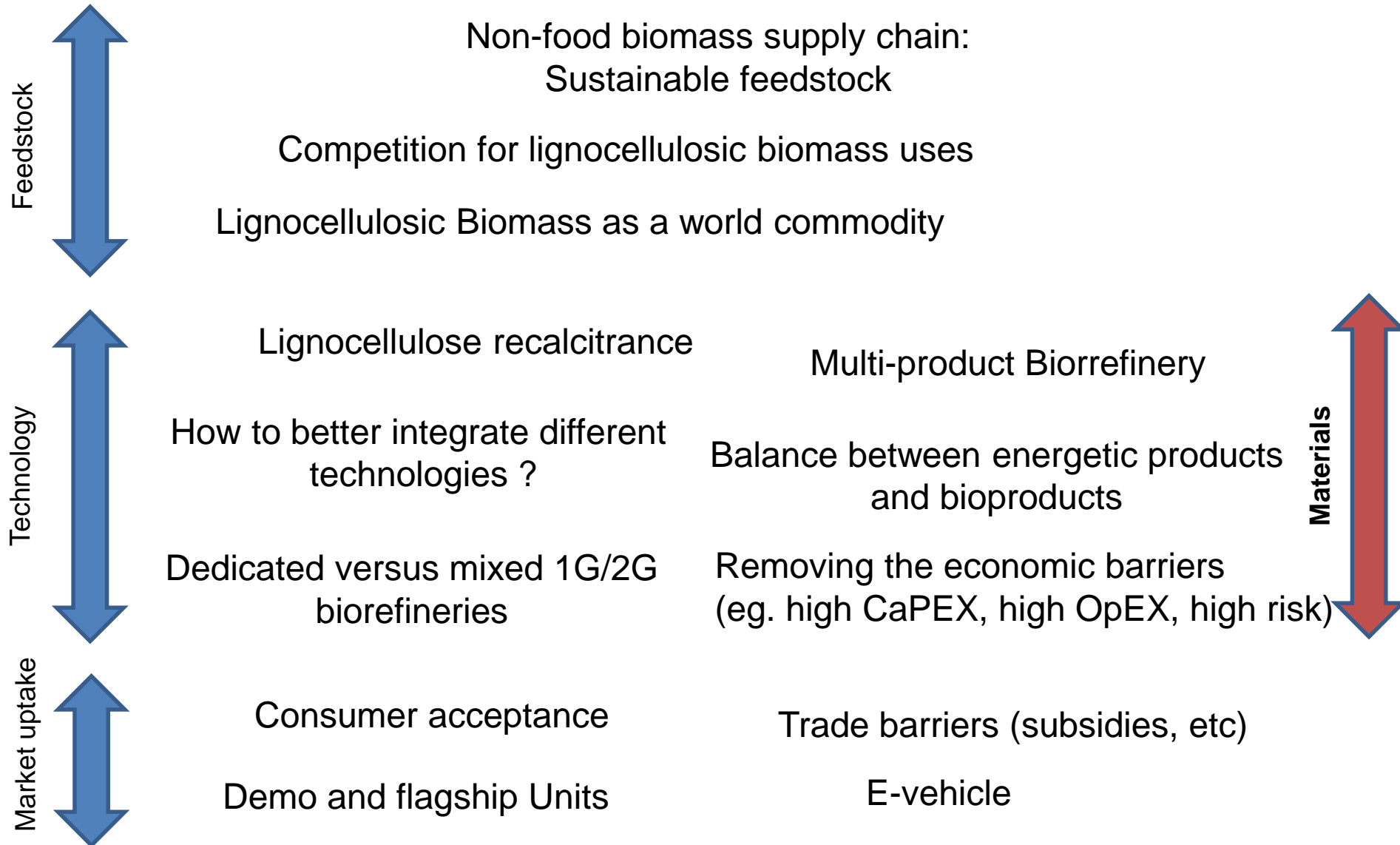
Competition for lignocellulosic biomass uses

Lignocellulosic Biomass as a world commodity

# Lignocellulosic-based Biofuel Biorefineries: What are the challenges for next 5-10 yrs?

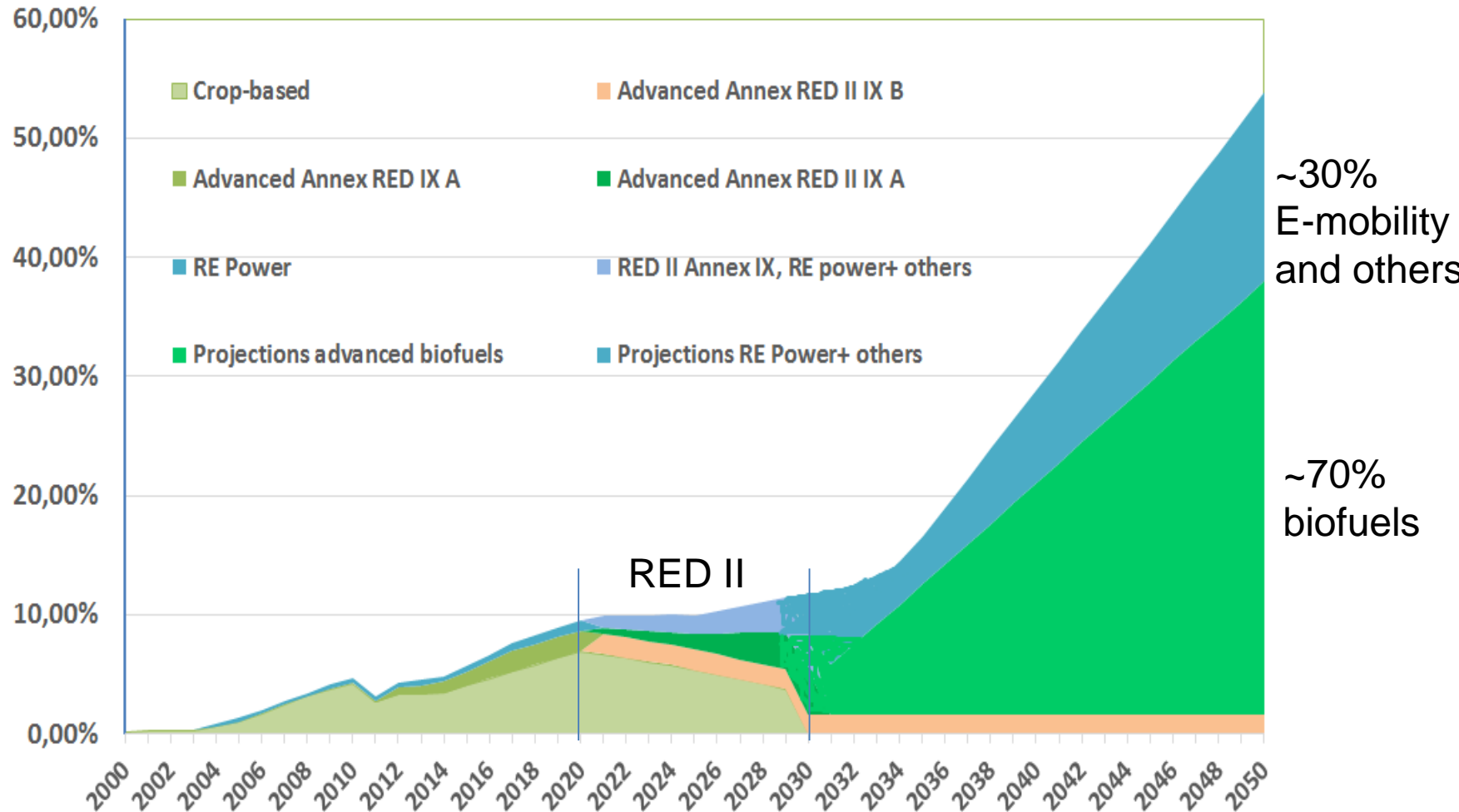


# Lignocellulosic-based Biofuel Biorefineries: What are the challenges for next 5-10 yrs?

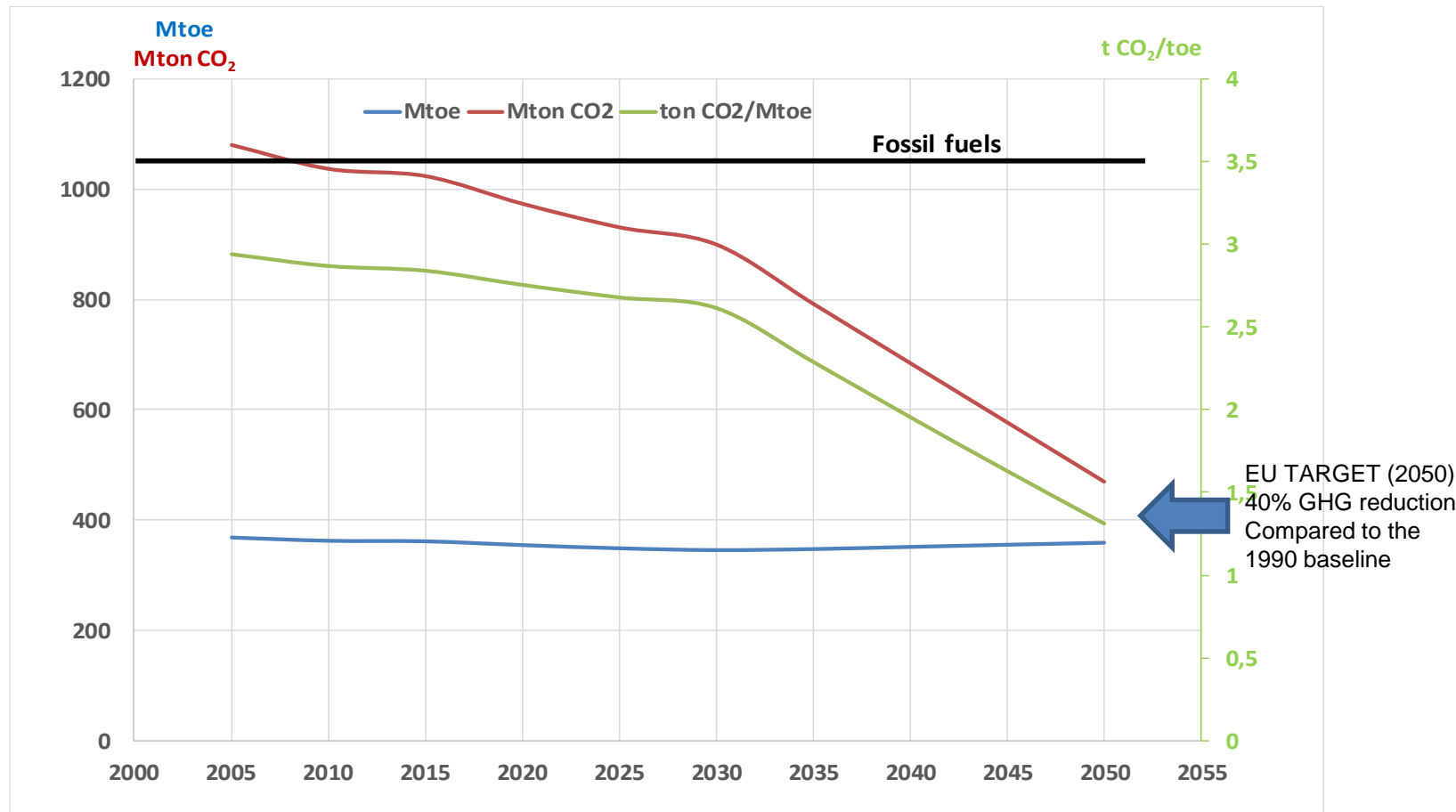


# 2050 Scenarios for the Transport Sector according to the the new proposed EC Policy (RED II) (30.11.2016, Winter Package)

Diagramrubrik

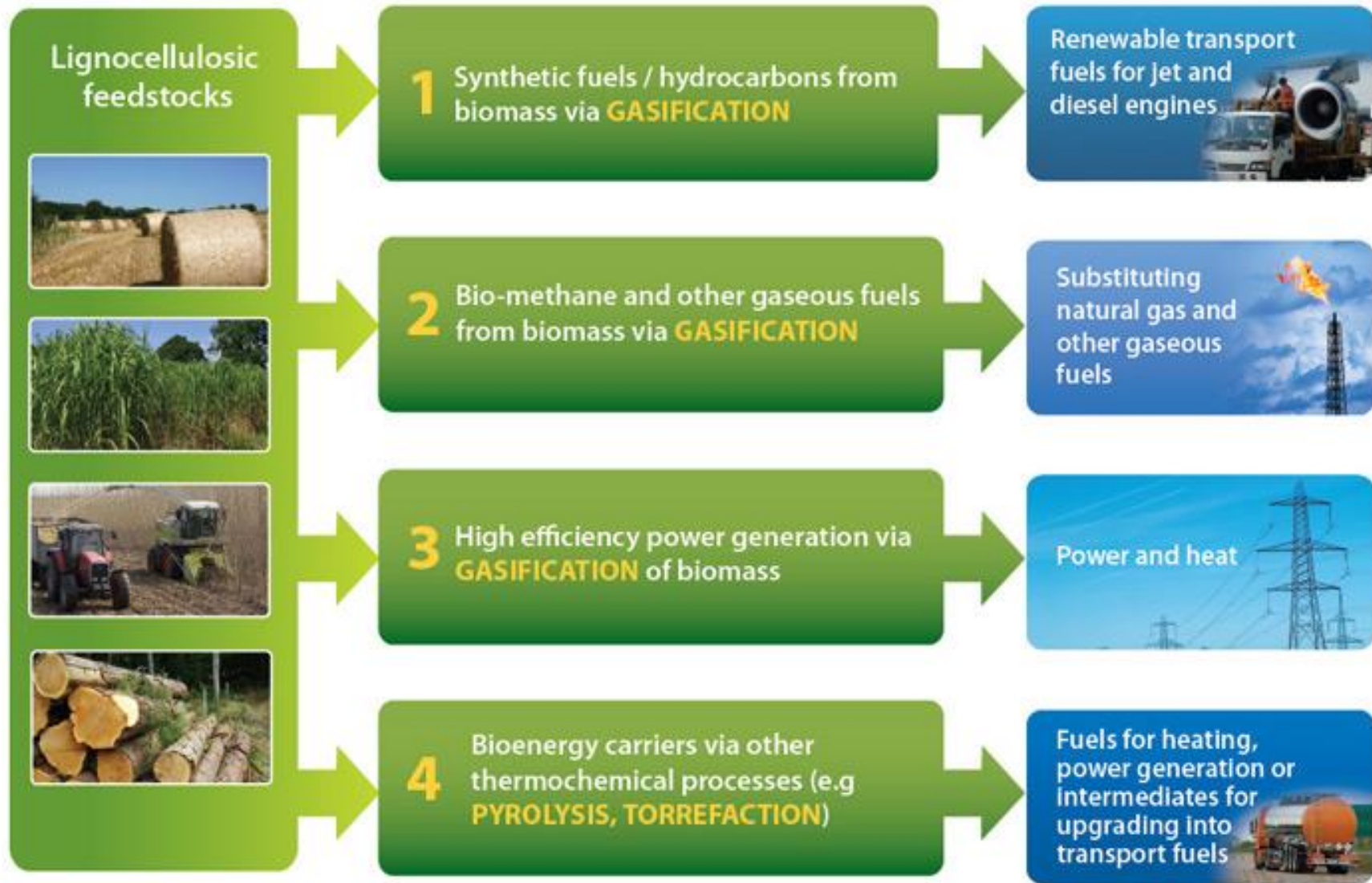


# 2050 Scenarios for the Transport Sector according to the the new proposed EC Policy (RED II) – GHG emissions reduction pattern (30.11.2016, Winter Package)



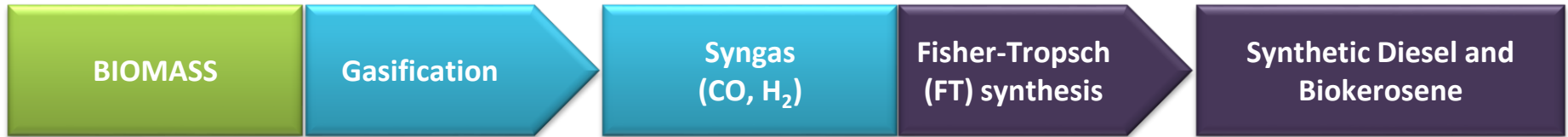
Source: Report "Building up the Future" (2017), Sub-Group of Advanced Biofuels do Sustainable Transport forum, 10 March, Edited por: Kyriakos Maniatis, Ingvar Landalv, Lars Waldheim, Eric van den Heuvel and Stamatis Kalligeros, DG ENER, EC, Brussels

# SET PLAN: The Seven Advanced Value Chains from Biomass for Energy (I)

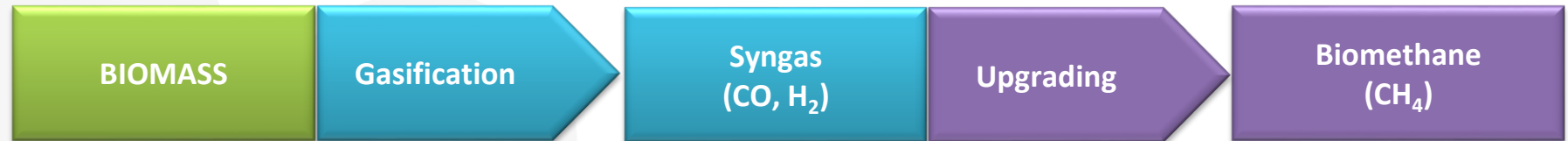


# Thermochemical Processing of Biomass

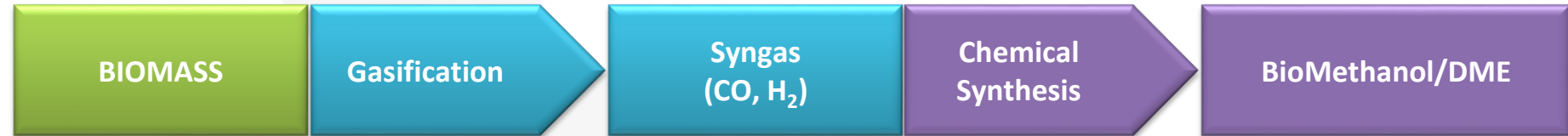
## Biomass-to-Liquid (BtL)



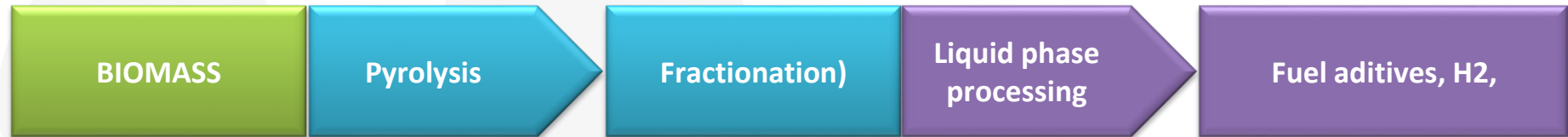
## Biomethane



## Methanol/DME

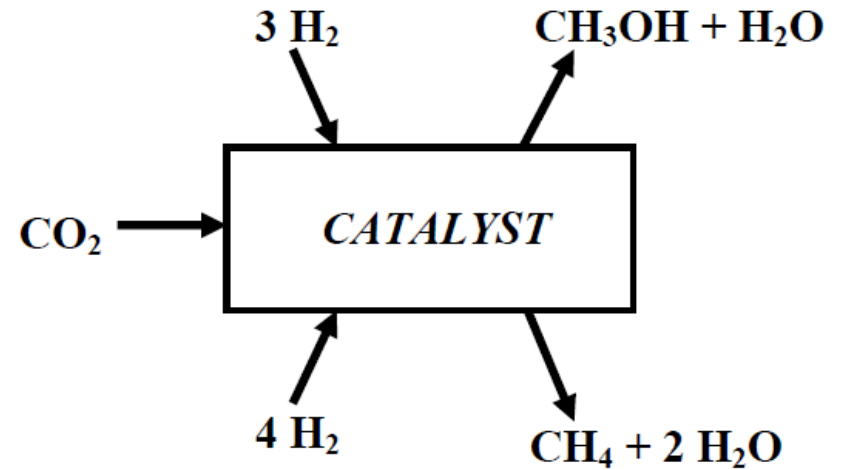


## Fuel additives, H<sub>2</sub>



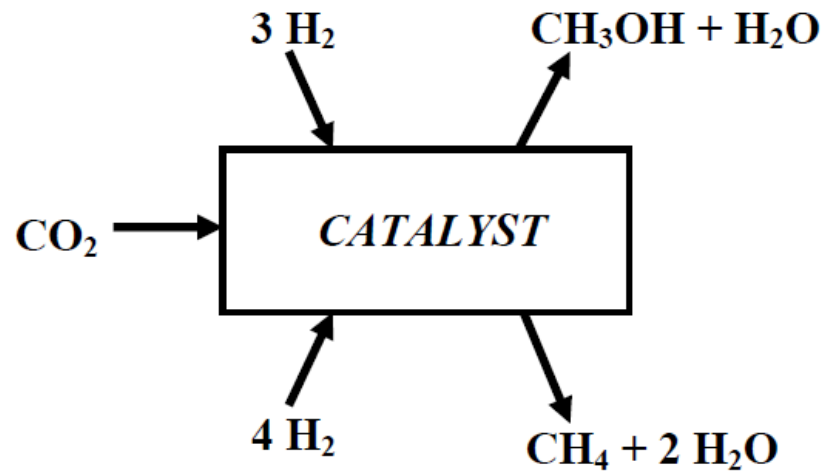
# Power-to-Gas & Power-to-Liquids

Routes for production of other Renewable Fuels of non-biological origin)

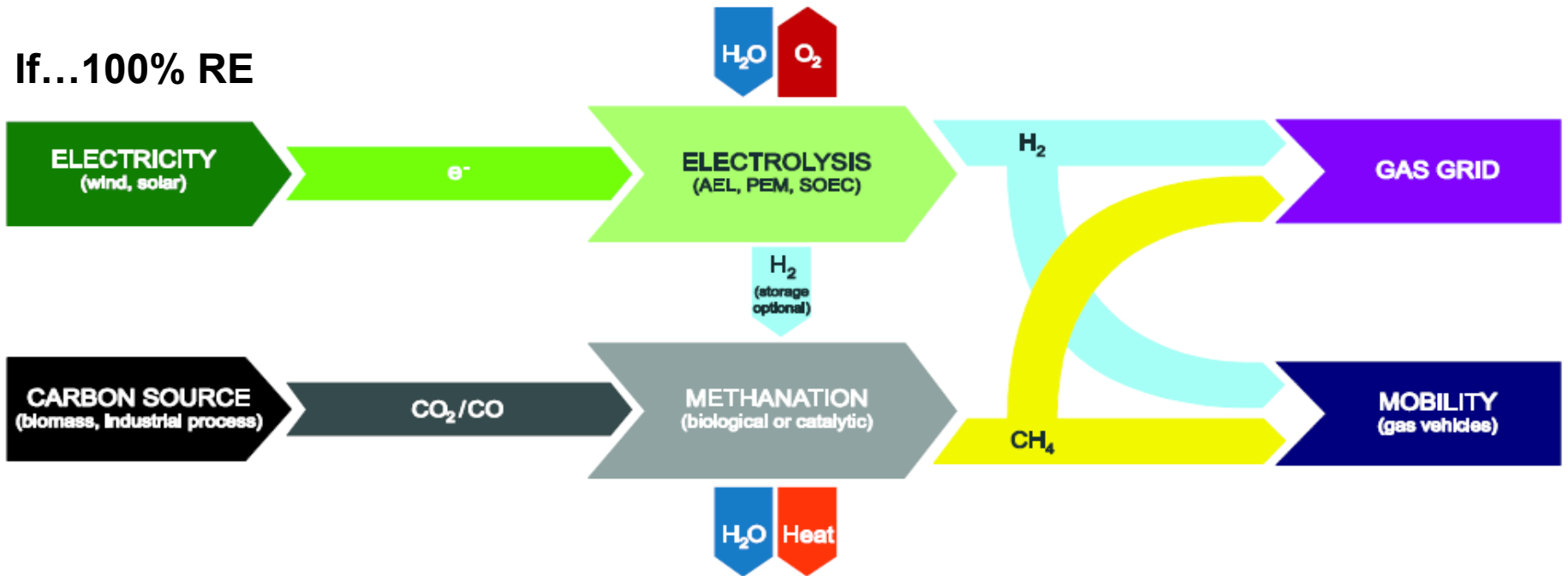


# Power-to-Gas & Power-to-Liquids

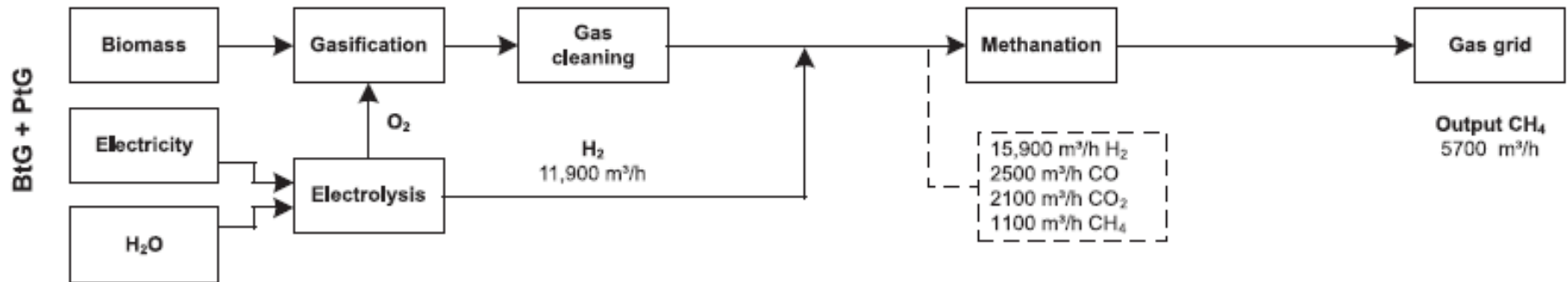
- Two steps:
1. H<sub>2</sub>O electrolysis using the RE surplus for H<sub>2</sub> production
  2. CO<sub>2</sub> as carbon source for Fuel synthesis (Methane) or (Methanol/DME)



If... 100% RE



# Mixed Conversion Processes: BtG + PtG

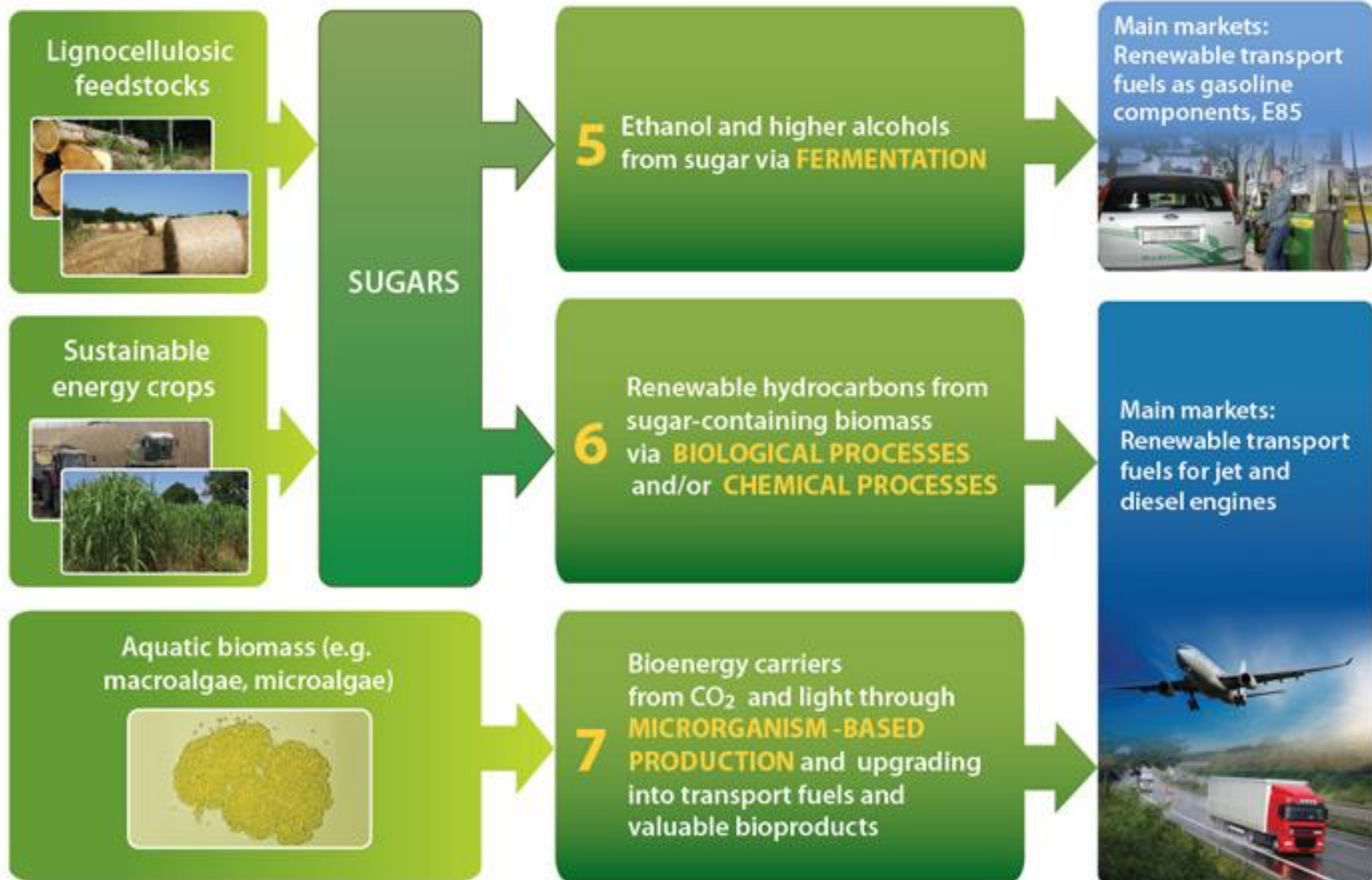


Source: Gotz et al (2016) *Renewable Energy*, 85, 1371-1390

## Main Bottlenecks:

- Electricity should be 100% renewable (and preferably, E-surplus)
- Necessary to develop fuel cell technology to improve water electrolysis technology
- Methanation reaction needs better “poison-insensitive” catalysts and should withstand with high temperature variations over a long operation period
- Hydrogen storage is also an issue (needs suitable tank reactors) unless it is injected into a NG grid (4% max.)

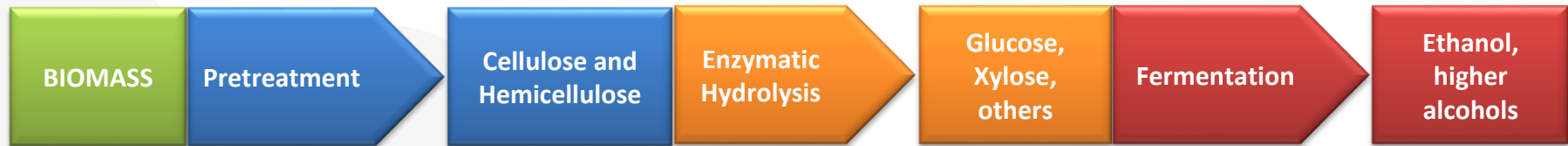
# SET PLAN: The Seven Advanced Value Chains from Biomass for Energy (II)



# (Bio)chemical Processing of Biomass

## Lignocellulosic materials, MSW, Other organic wastes

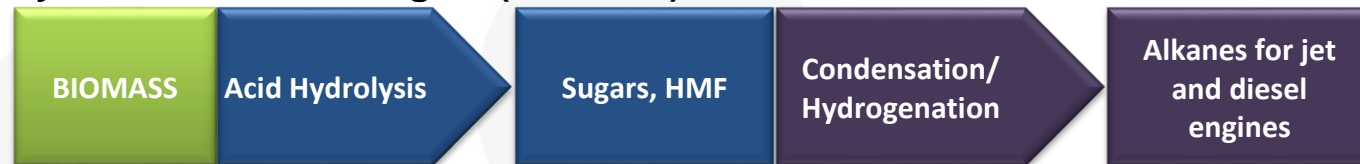
### Lignocellulosic ethanol, higher alcohols



### Hydrocarbons from sugars (biochemical)



### Hydrocarbons from sugars (chemical)

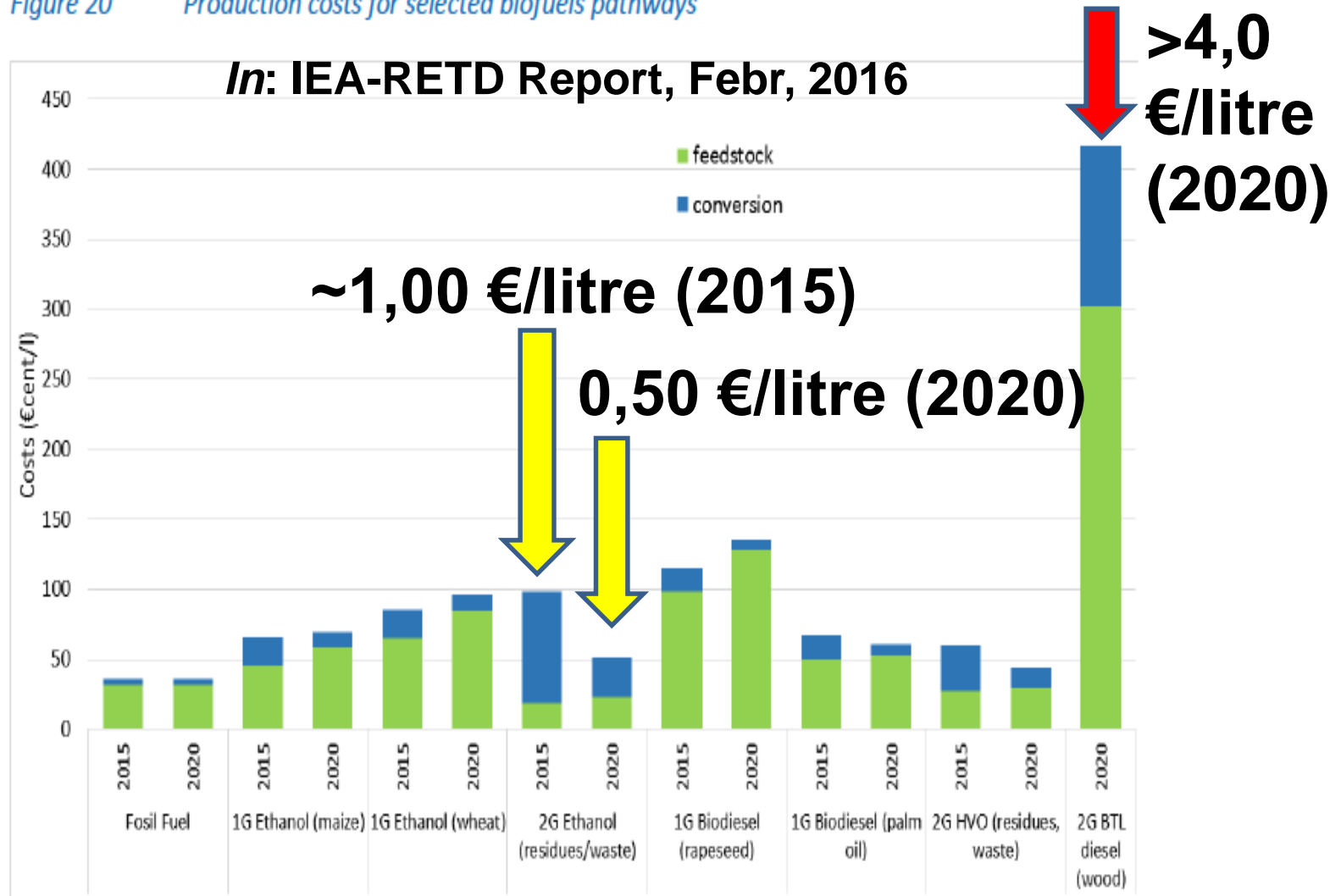


### Algae



# Production Costs Benchmarking of Thermochemical vs. Biochemical conversion of Biomass for Advanced Biofuels

Figure 20 Production costs for selected biofuels pathways



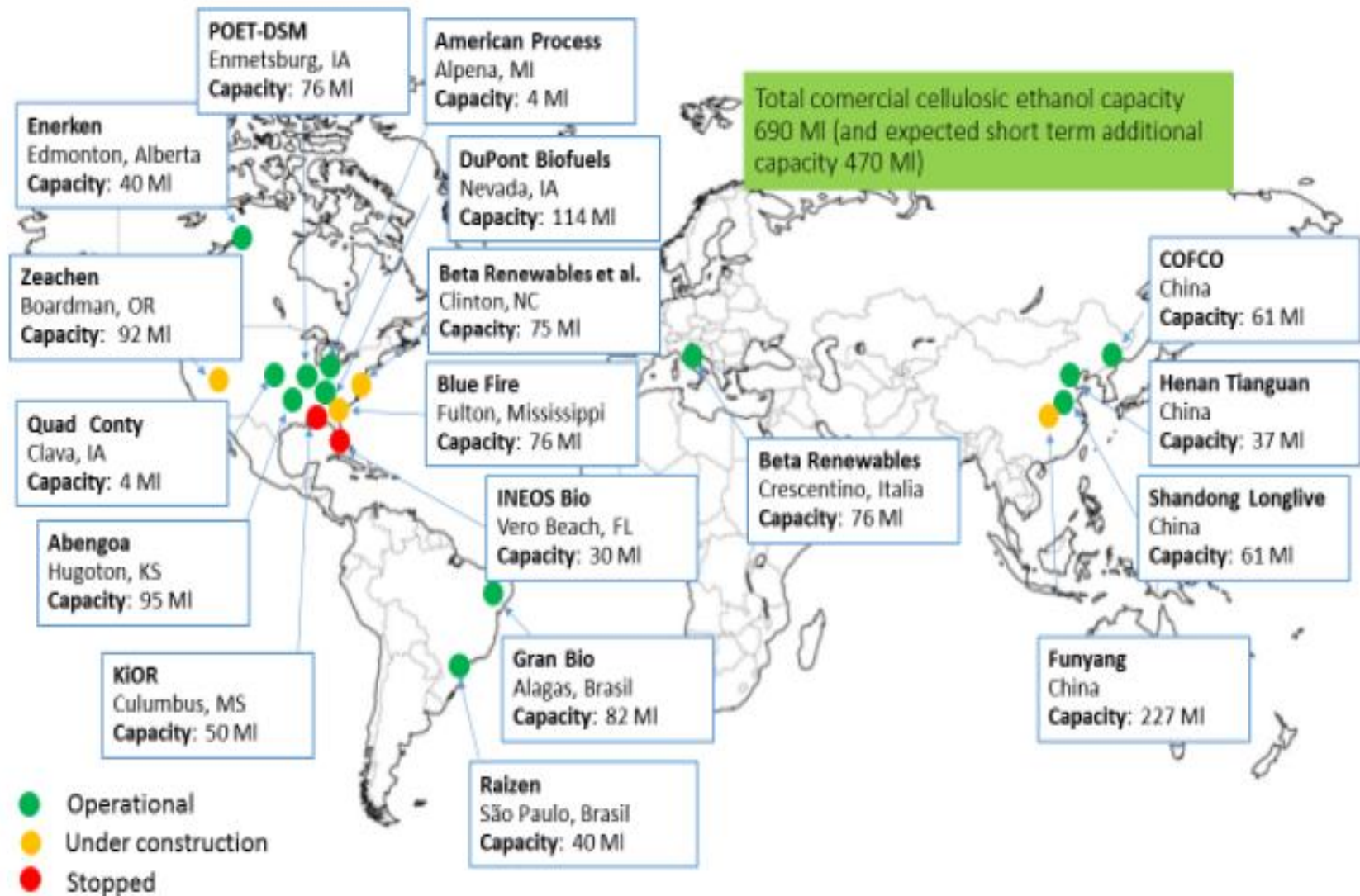
Source: Adapted from Festel et al. (2014); data given for 50 €/bbl crude oil price

# **Biorefineries in the World**

***Current Status of***

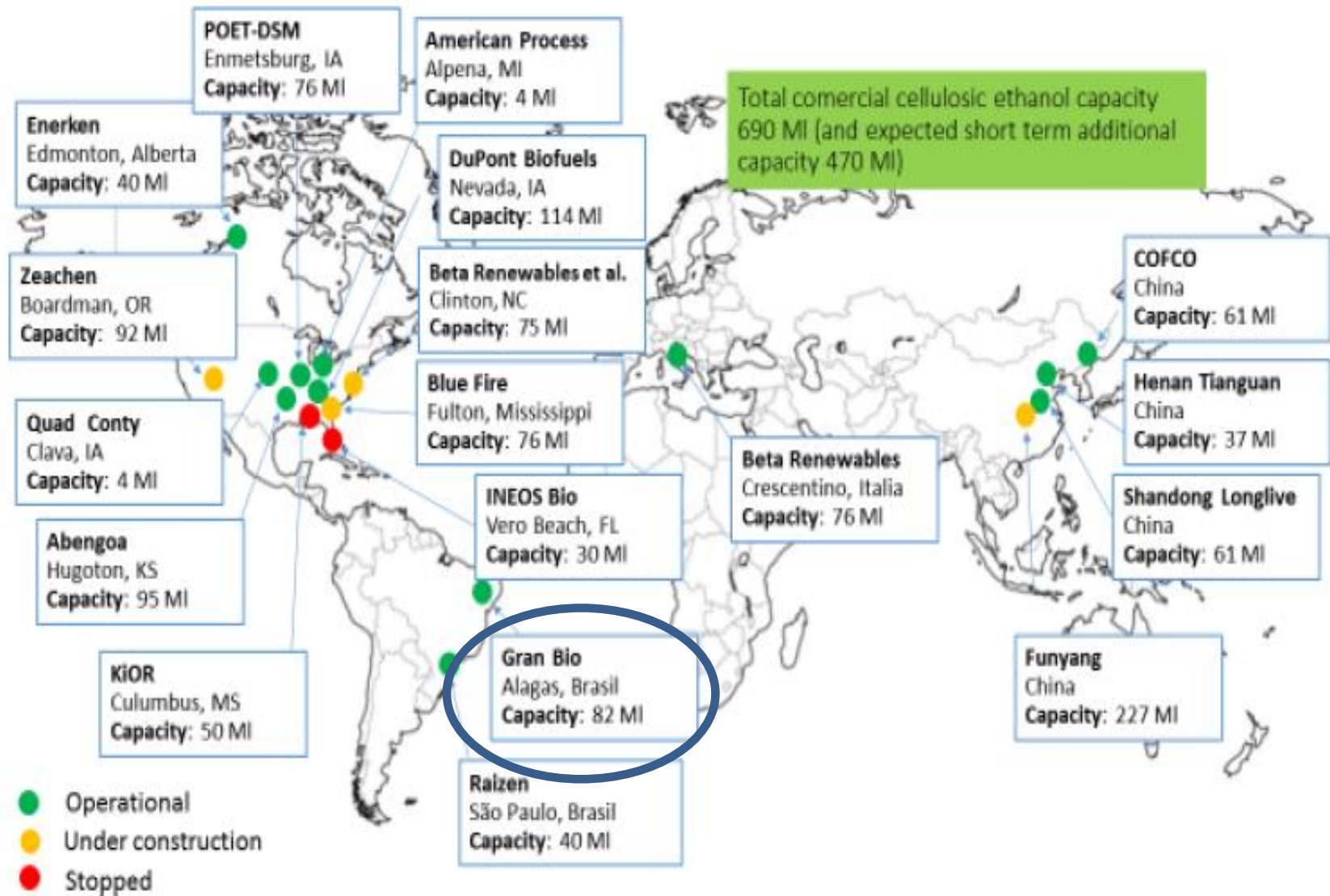
**Commercial & Demo  
Advanced Bioethanol Plants**

# World Panorama of Advanced Bioethanol Plants



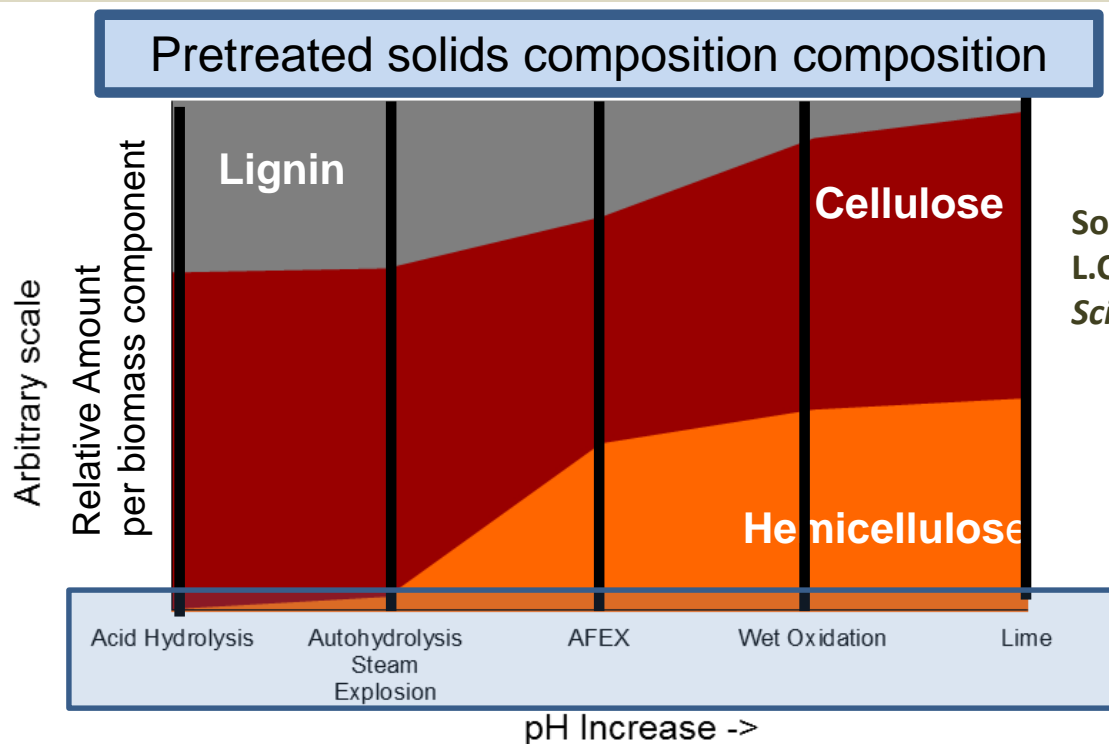
Source: Mizutani (2015) and Hailong (2015); data given for annual production capacity of 2G ethanol

# World Panorama of Advanced Bioethanol Plants



Source: Mizutani (2015) and Hailong (2015); data given for annual production capacity of 2G ethanol

# LIGNOCELLULOSIC BIOMASS PRE-TREATMENTS: Current state-of-the-art



Source: Carvalheiro, F., Duarte, L.C., Gírio, F. M. (2008). *J. Scientific & Ind. Res.*, 67, 849-864

## Key R&D Areas

- No standard pretreatment technology available for any type of feedstock biomass
- Steam explosion is the main technology at Demo and Commercial plants
- clean biomass pressurization is a bottleneck
- Effective one-pot pretreatment separation of three main streams are still lacking
- Reduce the energy demand for biomass pretreatments is an issue
- Recovery of high quality lignin during pretreatment shall be a major advantage (not attained with steam explosion or similar pretreatments)
- Better overall integration “Pretreatment-Fermentation-Separation” is necessary

# Biochemtex/BetaRenewables

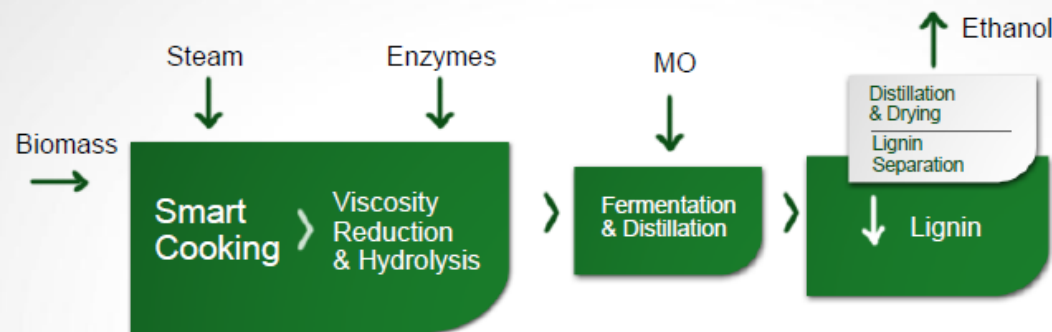
- First **commercial** cellulosic bioethanol plant in Europe has been deployed in Crescentino, Italy, since end 2013

**Raw material:** 270.000 ton/year cereals straw, (in future: *arundo donax*)

**Product:** Ethanol (60.000 ton/year), biogas, H<sub>2</sub>, lignin for energy

**Production process:** uncatalyzed two-stage steam explosion, enzymatic hydrolysis and co-fermentation of C<sub>5</sub> and C<sub>6</sub>

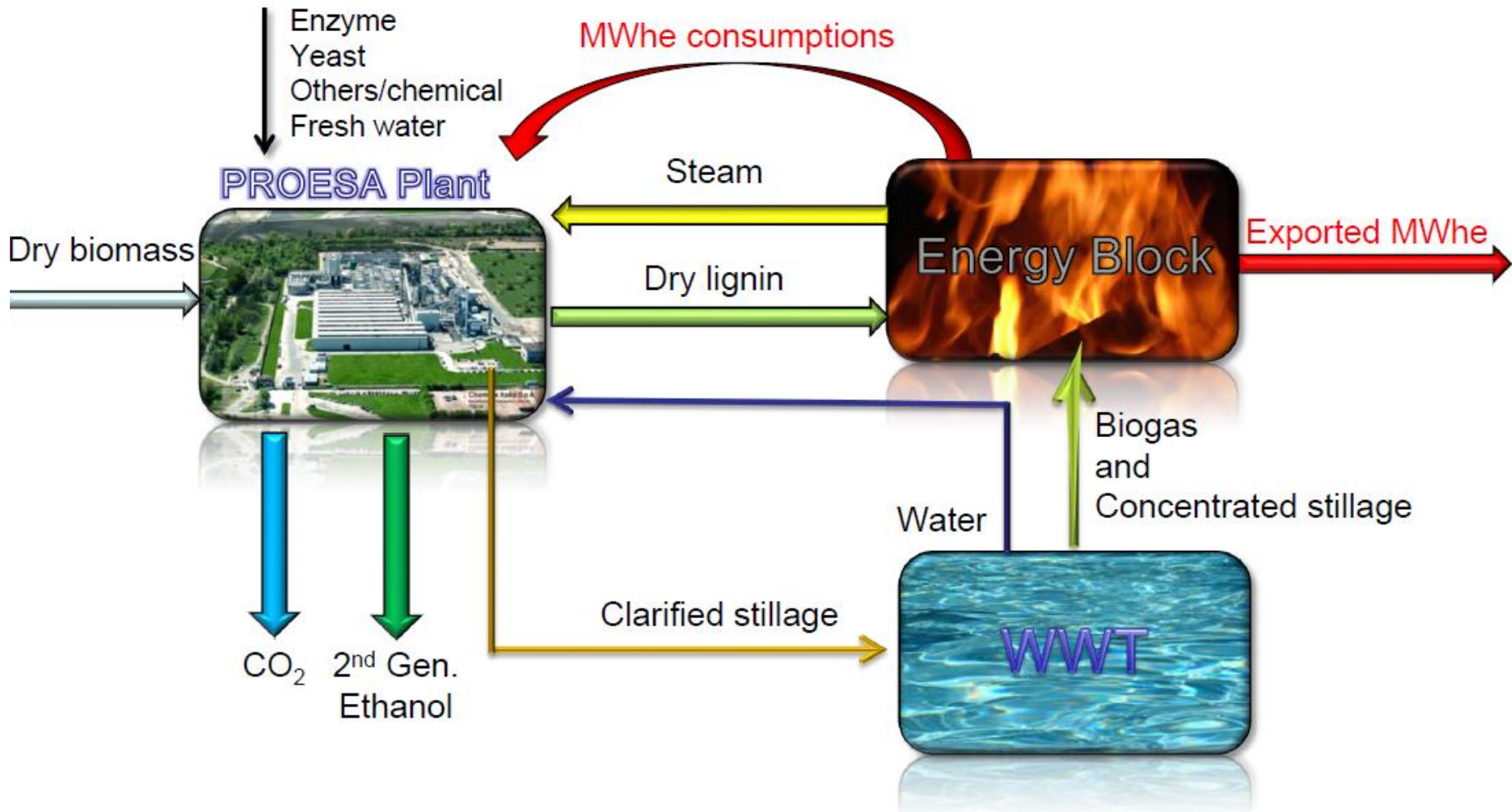
## Proesa® technology – Phase 1



Biochemtex/BetaRenewables



# 2G Ethanol plant configuration




# PROESA® Cellulosic Sugar Technology

Pre-Treatment Section



Enzymatic Hydrolysis Section



**BIOFUELS**

- Ethanol
- Bio-Jet
- Butanol 

**BIOCHEMICALS**

- Fatty Alcohols 
- 1,4 Butanediol 
- Farnasene
- Acrylic Acid
- Succinic Acid
- Others

**LIGNIN CHEMICALS**

- Phenols
- Xylene
- Terephthalic Acid



# Inbicon/Dong Energy

**Local:** Kalundborg (Denmark) – Demo plant

**Start-up:** 2009

**Raw material:** 30.000 ton/year wheat straw

**Product:** Ethanol (4.300 ton/year), C<sub>5</sub> molasses, lignin for energy

**Production process:** Hydrothermal pre-treatment, enzymes from Novozymes, enzymatic hydrolysis and co-fermentation of C<sub>6</sub> (phase 1); C<sub>5</sub>+C<sub>6</sub> co-fermentation (phase 2). Stand-alone plant.

Demo

[www.inbicon.com](http://www.inbicon.com)



# Inbicon version 1 – C6 fermentation

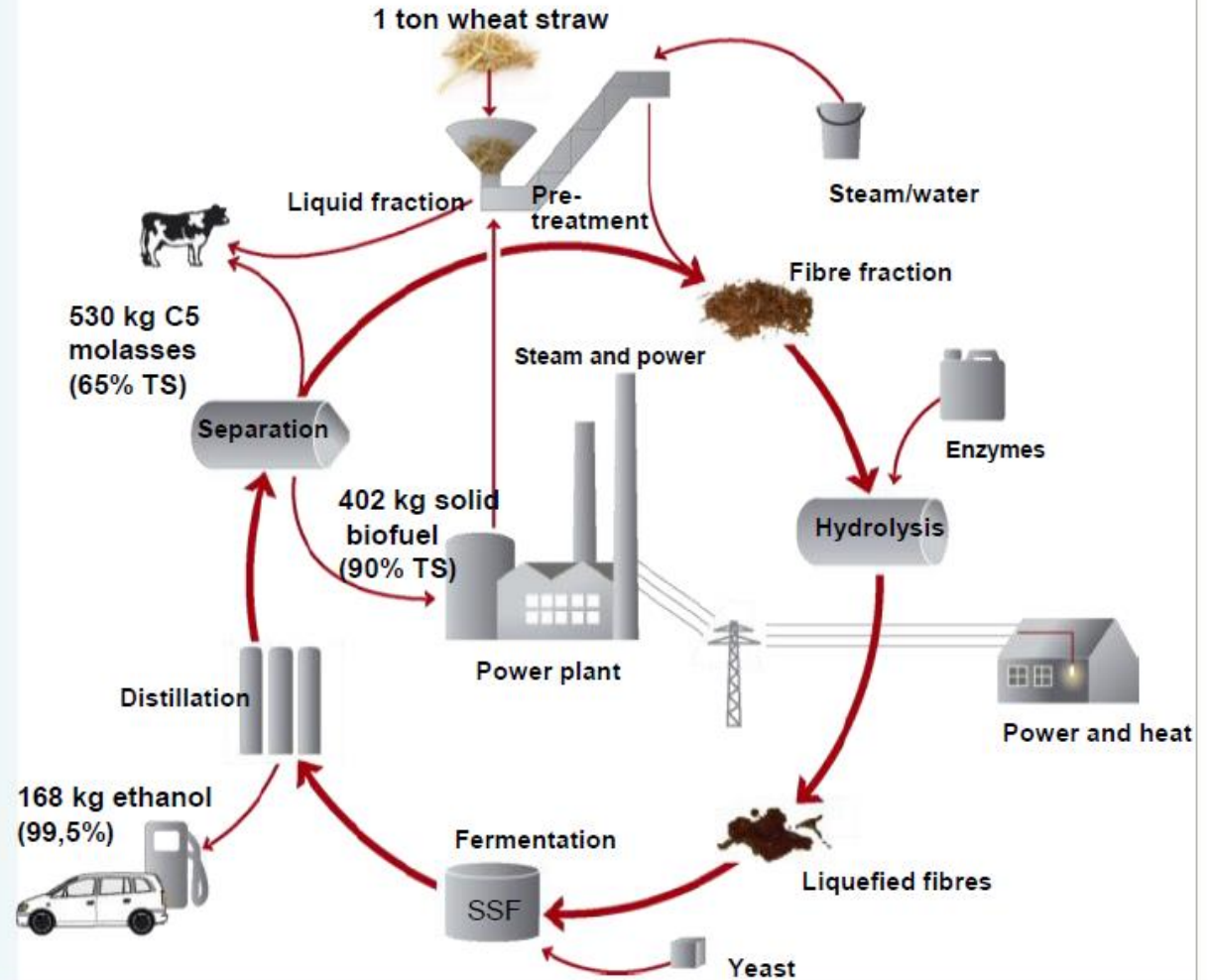
## Inbicon Concepts

- Biomass
- Water
- Enzymes
- Yeast



- Bioethanol
- Solid Biofuel
- C5 Molasses

Typical yield of 200 l  
etoh per ton of  
biomass dry matter



# Inbicon version 2 – C6+C5 mixed fermentation – proven in demo scale

## Inbicon Concepts

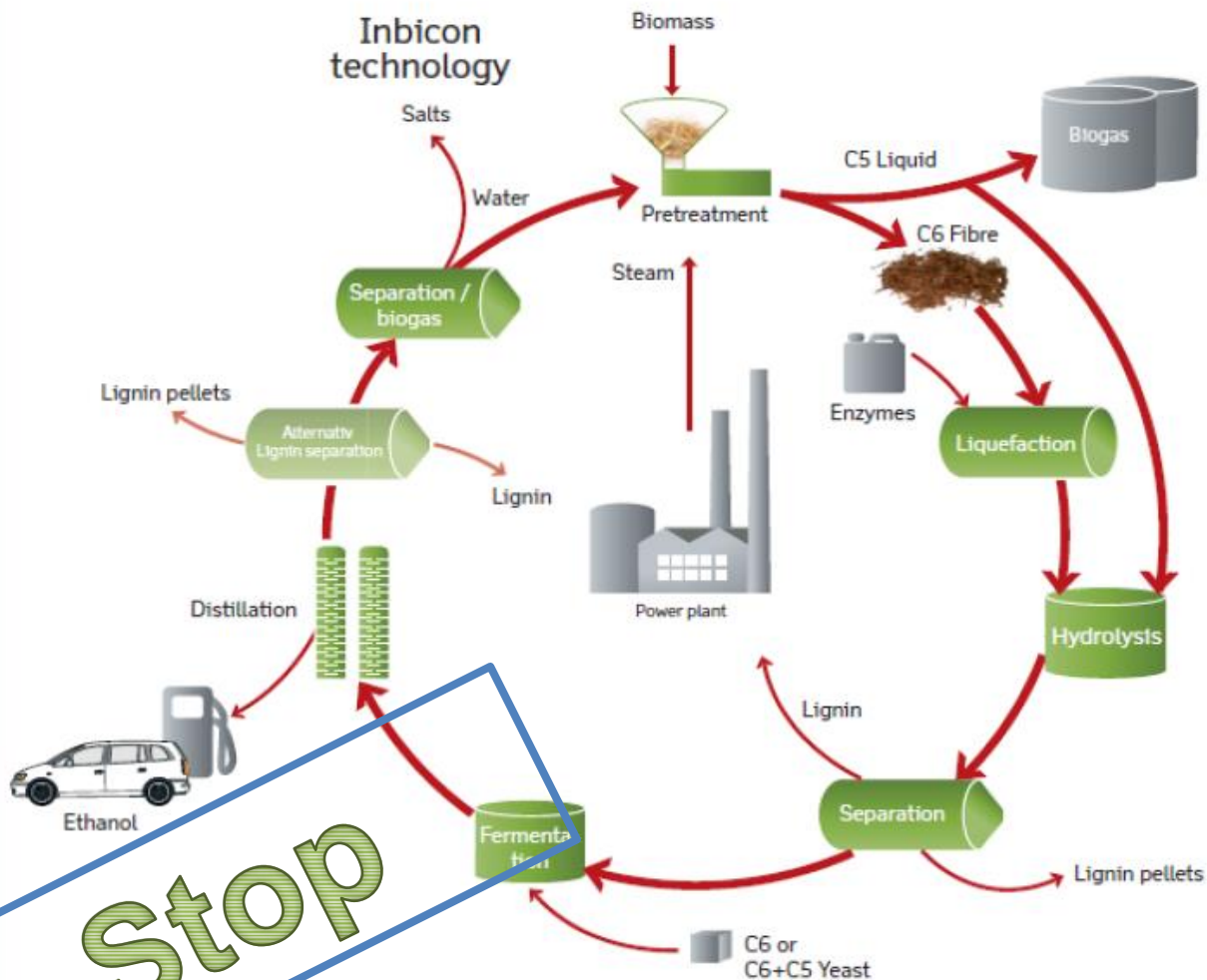
- Biomass
- Water
- Enzymes
- Advanced yeast (GMO)



- Bioethanol
- Solid Biofuel
- Vinasses

- Improving ethanol yield with 40% in comparison to Inbicon version 1

- Typical yield of 280-300 l etoh per ton of biomass dry matter



# Clariant (ex- Sud-Chemie)

**Local** : Straubing (Germany)

**Start-up**: 2012

**Raw material**: cereal straw, agricultural waste

**Product**: Ethanol (1.000 ton/year)

**Production process**: steam explosion pre-treatment, enzymatic hydrolysis and co-fermentation of C<sub>5</sub> and C<sub>6</sub>

Demo

[www.sunliquid.com](http://www.sunliquid.com)



# Sunliquid® technology

FEEDSTOCK

PRE-TREATMENT

HYDROLYSIS

FERMENTATION

CELLULOSIC ETHANOL



INTEGRATED ENZYME  
PRODUCTION

## Key features and advantages

- Integrated enzyme production
- Fermentation of C6 and C5 sugars into ethanol
- Feedstock and process specific enzymes
- Energy saving ethanol separation technology



# Sunliquid® technology –



sunliquid®

Cellulosic Sugars

sunliquid®

Cellulosic Ethanol

Catalytic conversion

Biotechnology

Catalytic conversion

Bio-based chemicals

➔ Biotechnology  
➔ Catalytic conversion

- sunliquid®**
- Sugar platform standalone
  - Development opportunities for biobased products
  - Extensive know-how in biocatalysis, strain optimization and heterogenous catalysis



# Borregaard Industries AS

**Local:** Sarpsborg (Norway)

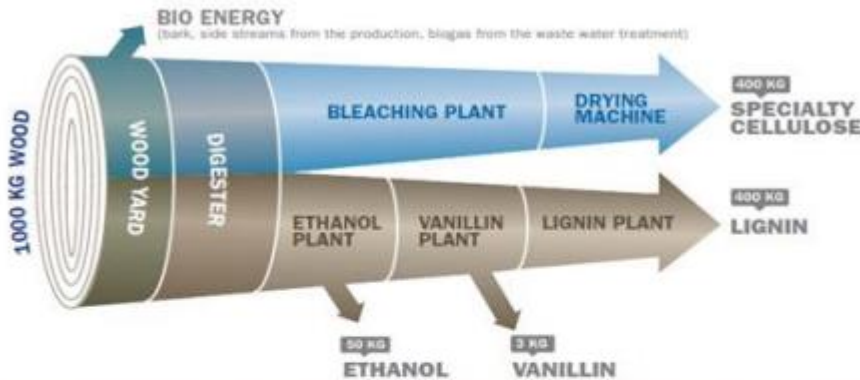
**BALI™ technology**

**Start-up:** 2013

**Raw material:** 50 kg/h; cereal straws, sugarcane bagasse

**Product:** Ethanol (110 ton ethanol/year or 22° ton sugars C<sub>5</sub>/C<sub>6</sub>/year; 200 ton/year specialized products of lignin)

**Production process:** Organosolv (BALI Technology); Biorefinery concept (chemical pretreatment, HE, Fermentation)



[www.borregaard.com](http://www.borregaard.com)

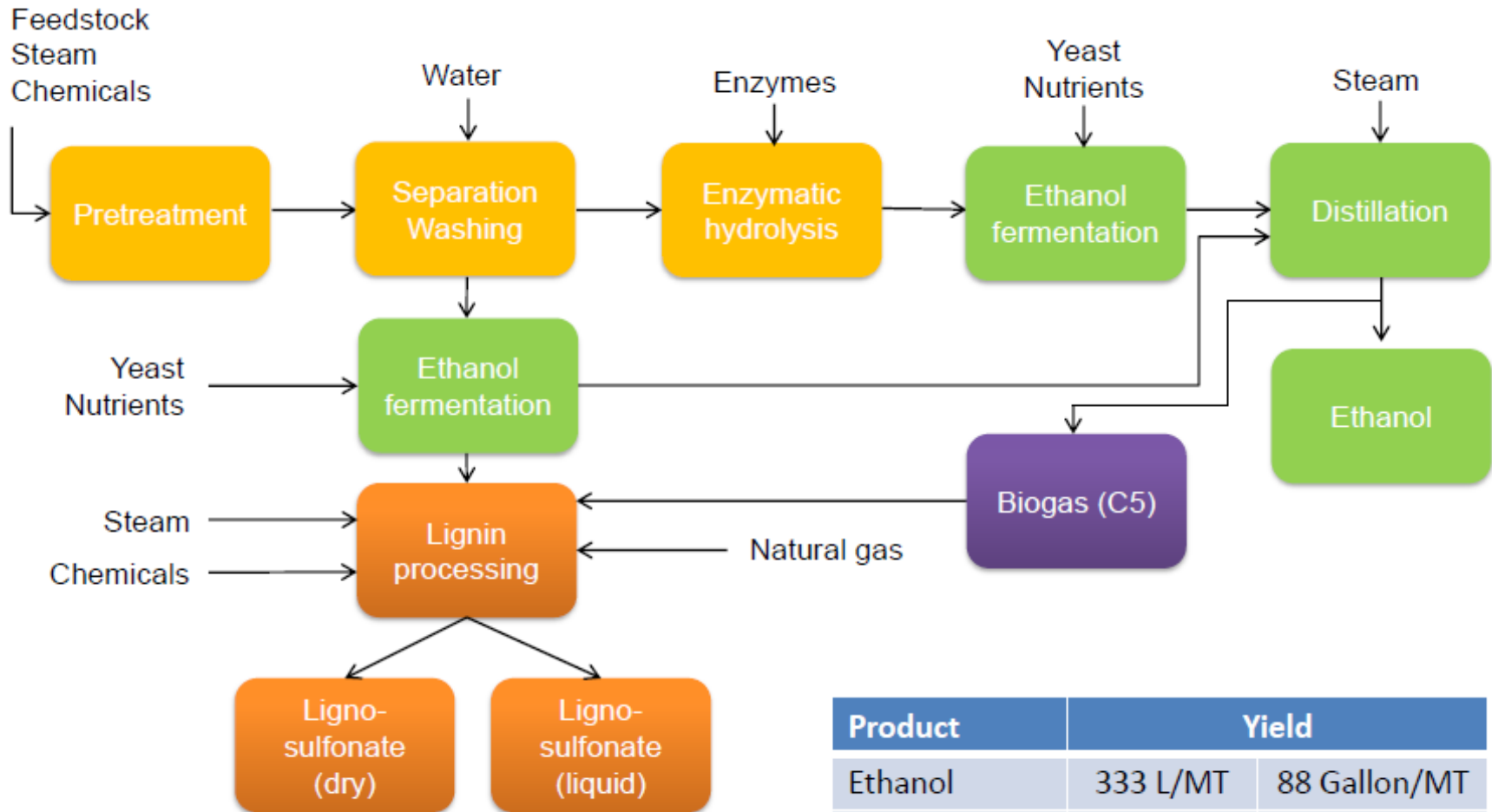
Cellulose	Lignin	Vanillin	Ethanol
Construction materials	Concrete additives	Food	Car care
Cosmetics	Animal feed	Perfumes	Paint/ varnish
Food	Dyestuff	Pharmaceuticals	Pharmaceutical industry
Tablets	Batteries		Bio Fuel
Textiles	Briquetting		
Filters	Mining		
Paint/ varnish			



Demo

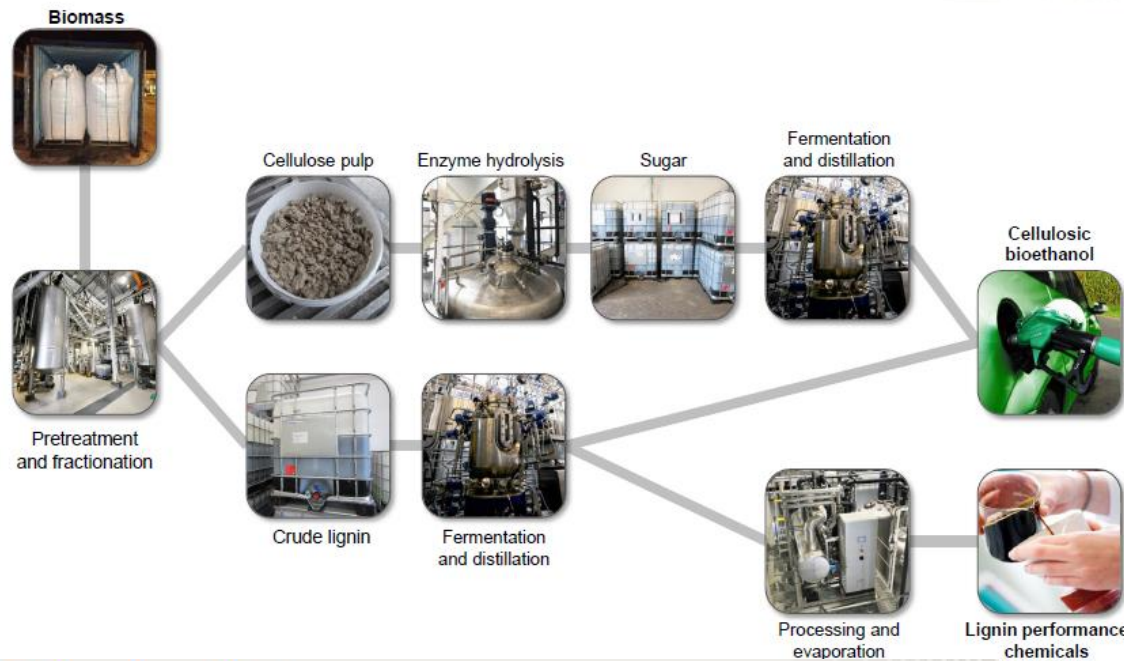
LNEG

# Example of a BALI™ softwood plant



Product	Yield	
Ethanol	333 L/MT	88 Gallon/MT
Lignin	450 kg/MT	

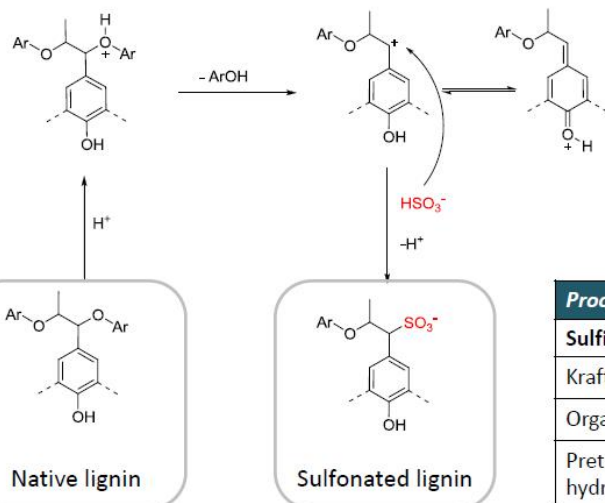
# BALI™ process in a nutshell



## Lignosulfonate as a dispersing agent



## Sulfonation of lignin



Process	Type of lignin
Sulfite pulping	Lignosulfonate
Kraft pulping	Alkali lignin
Organosolv pulping	Organosolv lignin
Pretreatment/hydrolysis	Hydrolysis lignin

## Lignosulfonates as water reducing agents in concrete

Strength of concrete inversely proportional to water/cement ratio  
 Lowered water content reduces workability of concrete  
 Addition of lignosulfonate reduces viscosity and hence improves flow and workability

Flow table test is a method to determine consistency of concrete.



Equipment; flow table, Abrams cone, waterbucket and broom.



The cone filled with concrete, prior to lifting.



The diameter of the resulting flow is measured

# DuPont

**Local:** Nevada - Iowa, USA

**Start-up:** 2015

**Raw material:** corn stover (350,000 ton/year)

**Product:** Ethanol (100,000 ton/year) + CHP (from lignin)

**Production process:** Diluted ammonia pretreatment, Enzymatic Hydrolysis, Bacterial Fermentation (recombinant *Z. mobilis*); no waste water (total water recycle)



Commercial

# DuPont Feedstock Collection Program

Contracting with more than 500 local farmers to gather, store and deliver over 349,000 dry tonnes of stover per year into the Nevada, Iowa facility.



# Poet/DSM (Project Liberty)

**Local:** Emmetsburg, Iowa, USA

**Start-up:** 2014

**Raw material:** corn stover (285,000 ton/year); 45-miles radius.

**Product:** Ethanol (60,000 ton/year) + biogas + CHP (from lignin)

**Production process:** Two-stage diluted acid pretreatment; C5+C6 fermentation; Co-location with an existing dry mil corn plant



Commercial

# GranBio (Brazil)

**Local:** São Miguel dos Campos - Alagoas, Brazil

**Start-up:** 2014

**Raw material:** sugar cane straw.

**Product:** Ethanol (65,000 ton/year) + electricity (from lignin and straw). Co-location with an existing 1G bioethanol plant from sugar cane.

**Production process:** Proesa™ uncatalysed steam explosion pretreatment; C5+C6 fermentation



Commercial



# Raízen (Brazil)

**Local:** Piracicaba - SP, Brazil

**Start-up:** 2015

**Raw material:** sugar cane bagasse and straw.

**Product:** Ethanol (32,000 ton/year) + electricity. Co-location with an existing 1G bioethanol plant from sugar cane.

**Production process:** logen's technology – acid-catalysed steam explosion,  
EH and Fermentation

Commercial



# Abengoa Bioenergy

**Local:** Hugoton, Kansas, US

**Start-up:** 2015 (Commercial)

**Raw material:** 350,000 ton/year corn stover (~15% available biomass in a radius of 50 miles)

**Product:** 75,000 ton Ethanol, biogas, 18 MWe and animal feed.

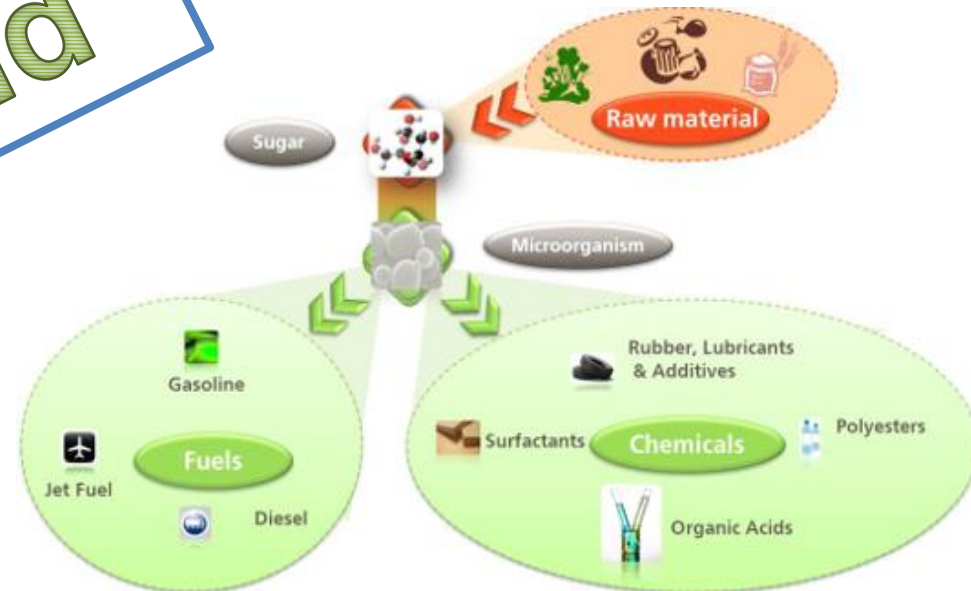
**Production process:**  $\text{H}_2\text{SO}_4$ -catalysed steam explosion pre-treatment, in situ enzyme production; enzymatic hydrolysis and co-fermentation of  $\text{C}_6$ . Stand-alone plant.

Commercial



Sold

Abengoa v2.0



# Abengoa Bioenergy (W2B)

**Local:** Babilafuente, Spain (2G Ethanol pilot plant retrofitting)

**Start-up:** 2011- 2015

**Raw material:** Organic fraction from MSW

**Product:** Ethanol (yield: 42% w/w)

In Sale?



Demo  
Plant



# Cost of 2G Bioethanol

- The main challenges of today's advanced biofuels projects in World are:
  - Competitiveness with either gasoline/diesel or with 1G (*necessary to address the key technological issues that influence final 2G biofuel price = "Technology improvement"*)
  - Price, logistics and availability of large scale biomass supply
  - New materials & technologies less energy-intensive and higher carbon conversion efficiency

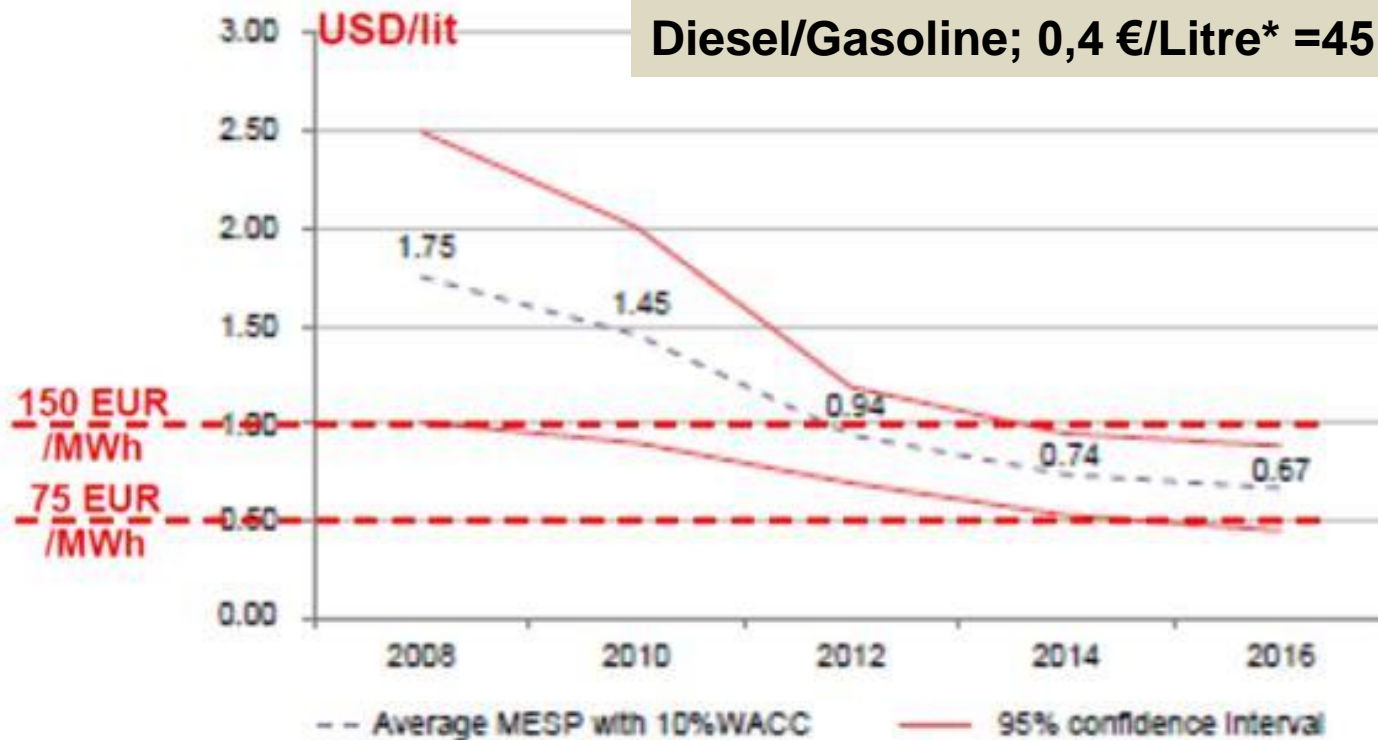
# Minimum 2<sup>nd</sup> generation ethanol selling price

(Source: Bloomberg's *Cellulosic ethanol costs: Surveying an industry*, March 2013)

Capital: 10% WACC  
Feedstock: 75 USD/mt (dry)

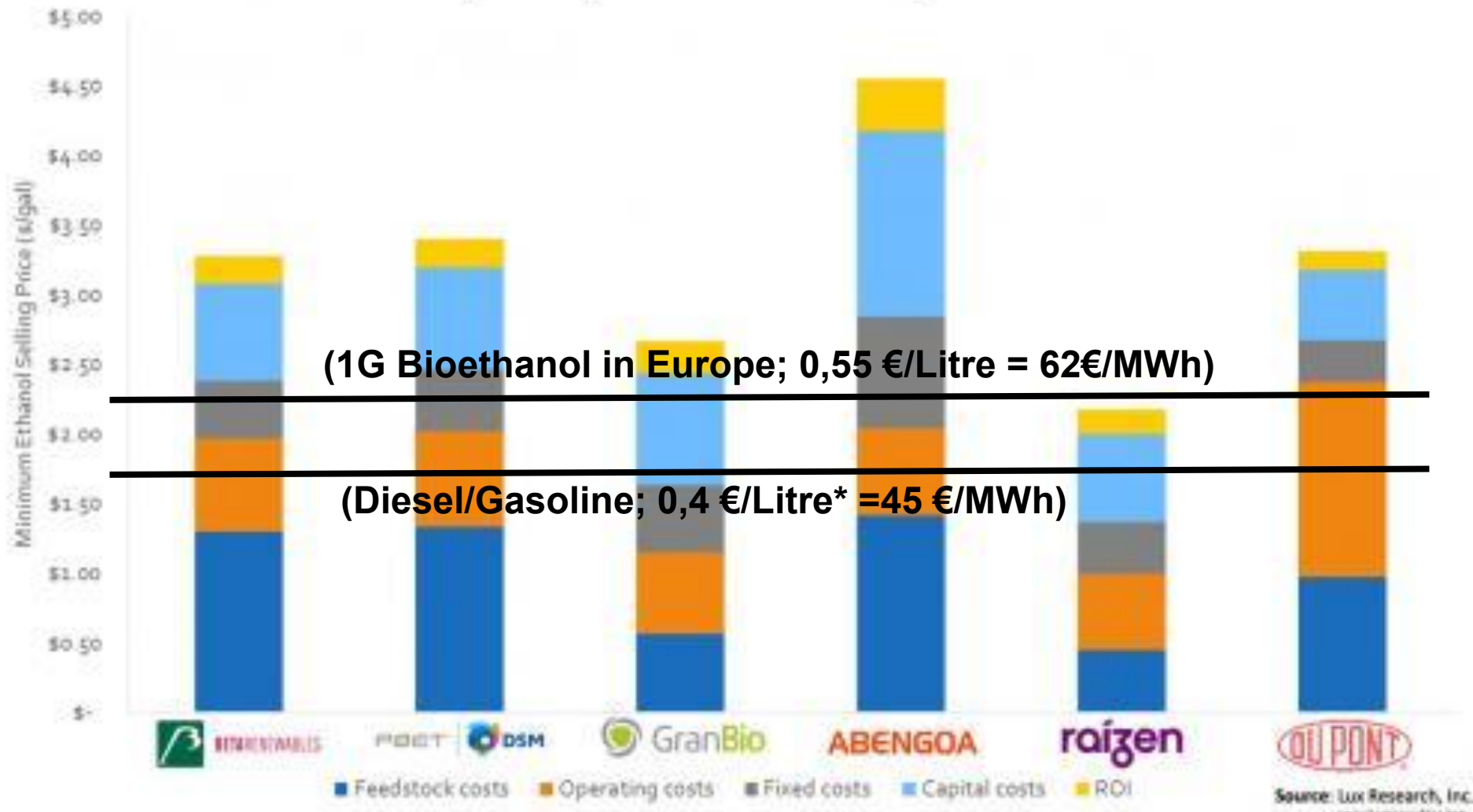
1G Bioethanol in Europe; 0,55 €/Litre = 62€/MWh

Diesel/Gasoline; 0,4 €/Litre\* = 45 €/MWh



Source: Bloomberg New Energy Finance Notes: the 95% confidence interval represents the area in which 95% of the survey participants' MESP's fell into – or two standard deviations from the mean; the MESP includes capex costs at 10% WACC; and feedstock costs are fixed at \$75 per dry tonne.

# Projected Major Cellulosic Ethanol Project Costs



(1G Bioethanol in Europe; 0,55 €/Litre = 62€/MWh)

(Diesel/Gasoline; 0,4 €/Litre\* = 45 €/MWh)

\* Considering 45 USD/bll crude oil

Source: Lux Research Inc (2015)

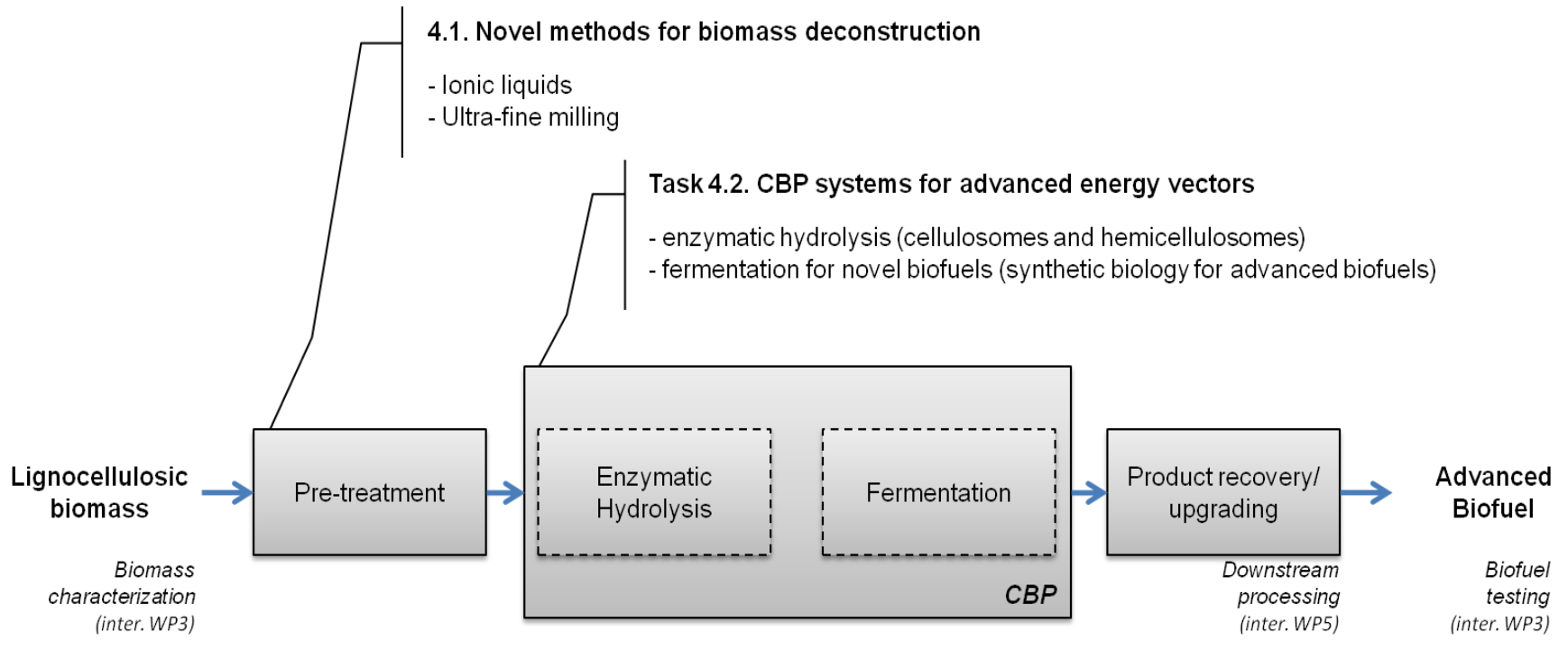
Source: Lux Research, Inc.  
www.luxresearchinc.com



# Why 2G Bioethanol (from biochemical routes) is still not cost-competing with 1G Bioethanol ?

- ❑ High CAPEX and OPEX costs
- ❑ Feedstock availability & supply (clean and at low-cost)
- ❑ Pretreatment and enzyme production are more costly and energy demand (and less sustainable in terms of GHG emissions) than “enzymatic hydrolysis + fermentation” steps.
- ❑ Lack of robust and sugars-fast fermentation by current 2G strains (1G strain consumes C6 sugars in 8 hours; the best 2G strains consumes LC sugars into 36-40 h)
- ❑ The non-fermentable component of biomass (LIGNIN) have to be burnt to supply the energy required for the overall plant energetic demand (*low energy-efficiency*)
- ❑ Quality of lignin

# Lignocellulosic Ethanol: ...the way forward for competing with 1G and fossils!

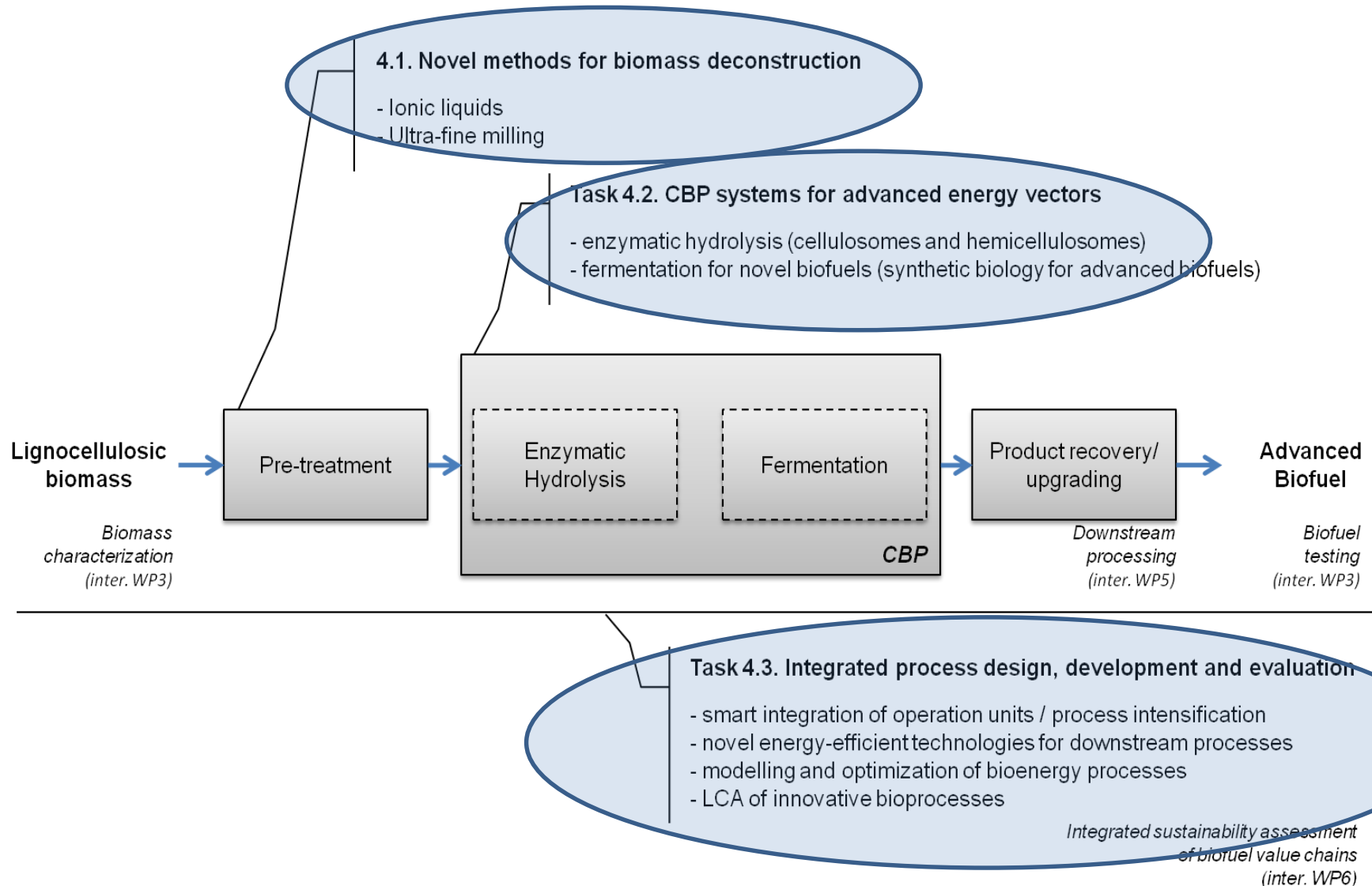


## Task 4.3. Integrated process design, development and evaluation

- smart integration of operation units / process intensification
- novel energy-efficient technologies for downstream processes
- modelling and optimization of bioenergy processes
- LCA of innovative bioprocesses

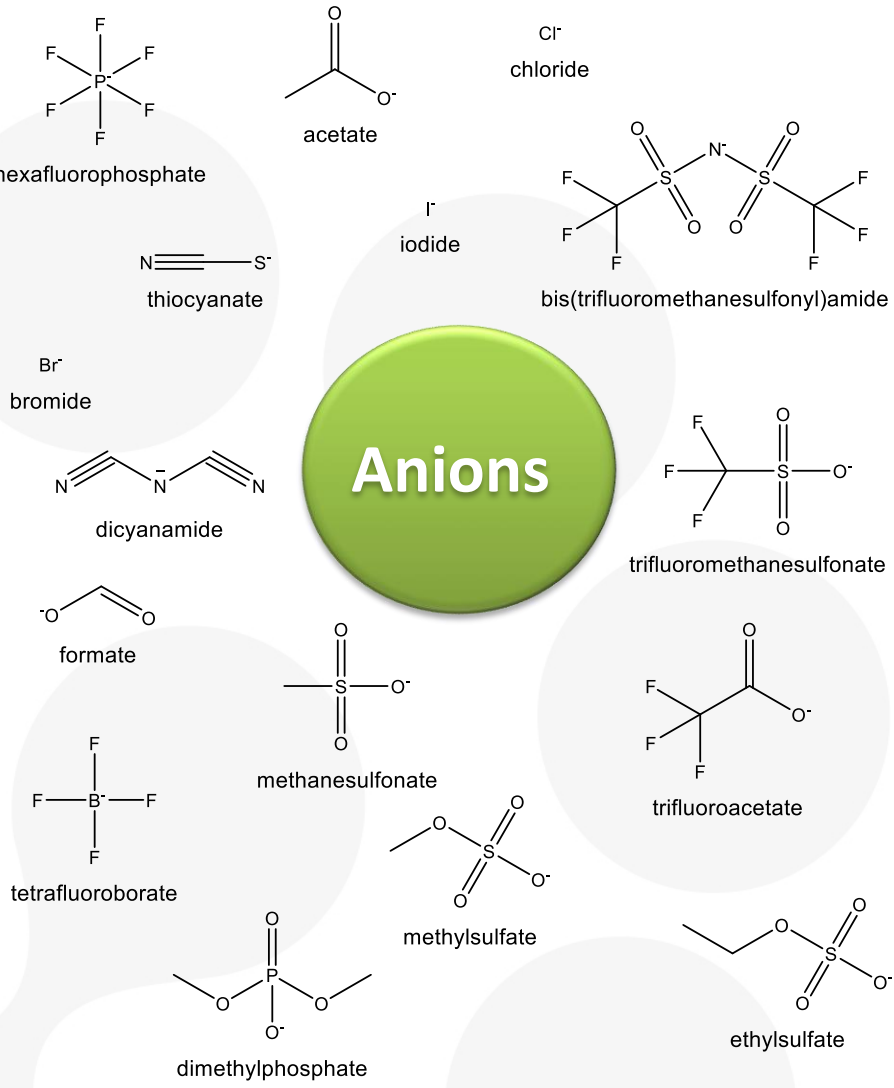
*Integrated sustainability assessment  
of biofuel value chains  
(inter. WP6)*

# Lignocellulosic Ethanol:...the way forward for competing with 1G and fossils

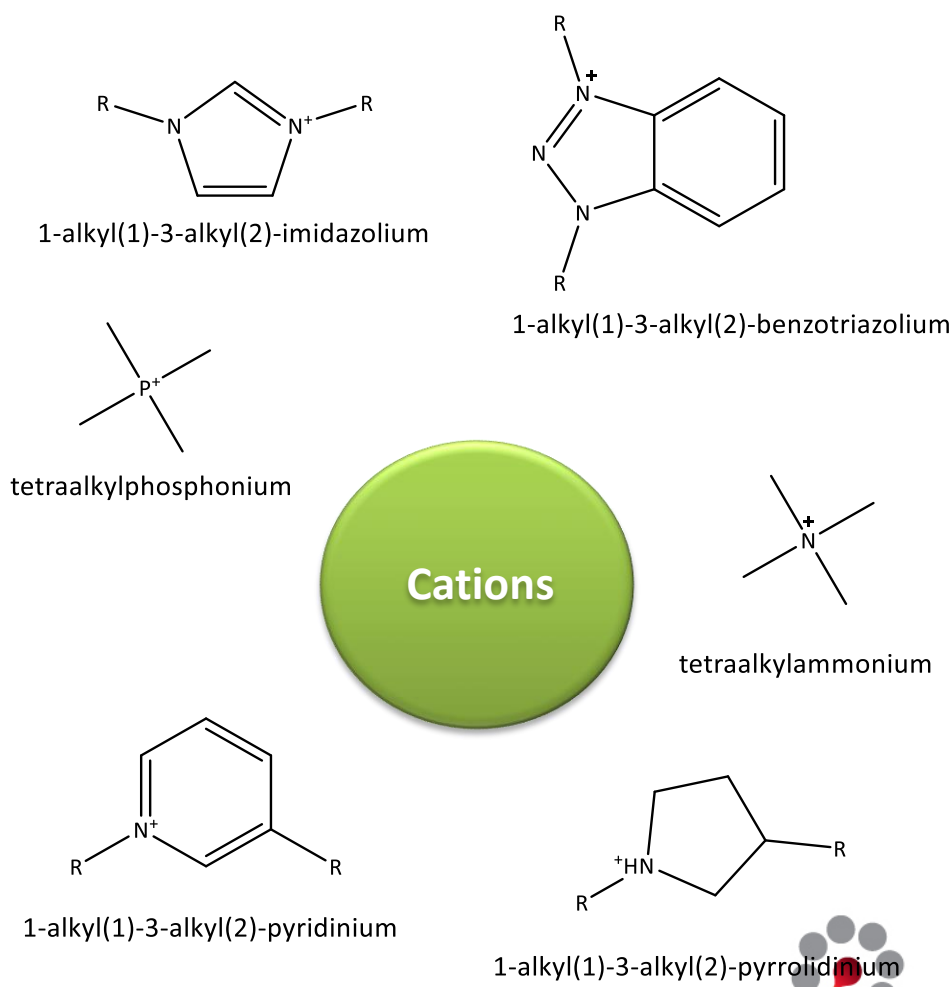


# Ionic liquids

## Anions



## Cations



# Ionic liquids

## General properties

- ❖ High polarity
- ❖ Negligible volatility
- ❖ Thermal stability
- ❖ High conductivity
- ❖ Large electrochemical window



## Tailored properties

- ❖ Density and Viscosity
- ❖ Solubility
- ❖ Lipophilicity and Polarity
- ❖ Acidity and Basicity

# Pre-treatment of biomass with ILs

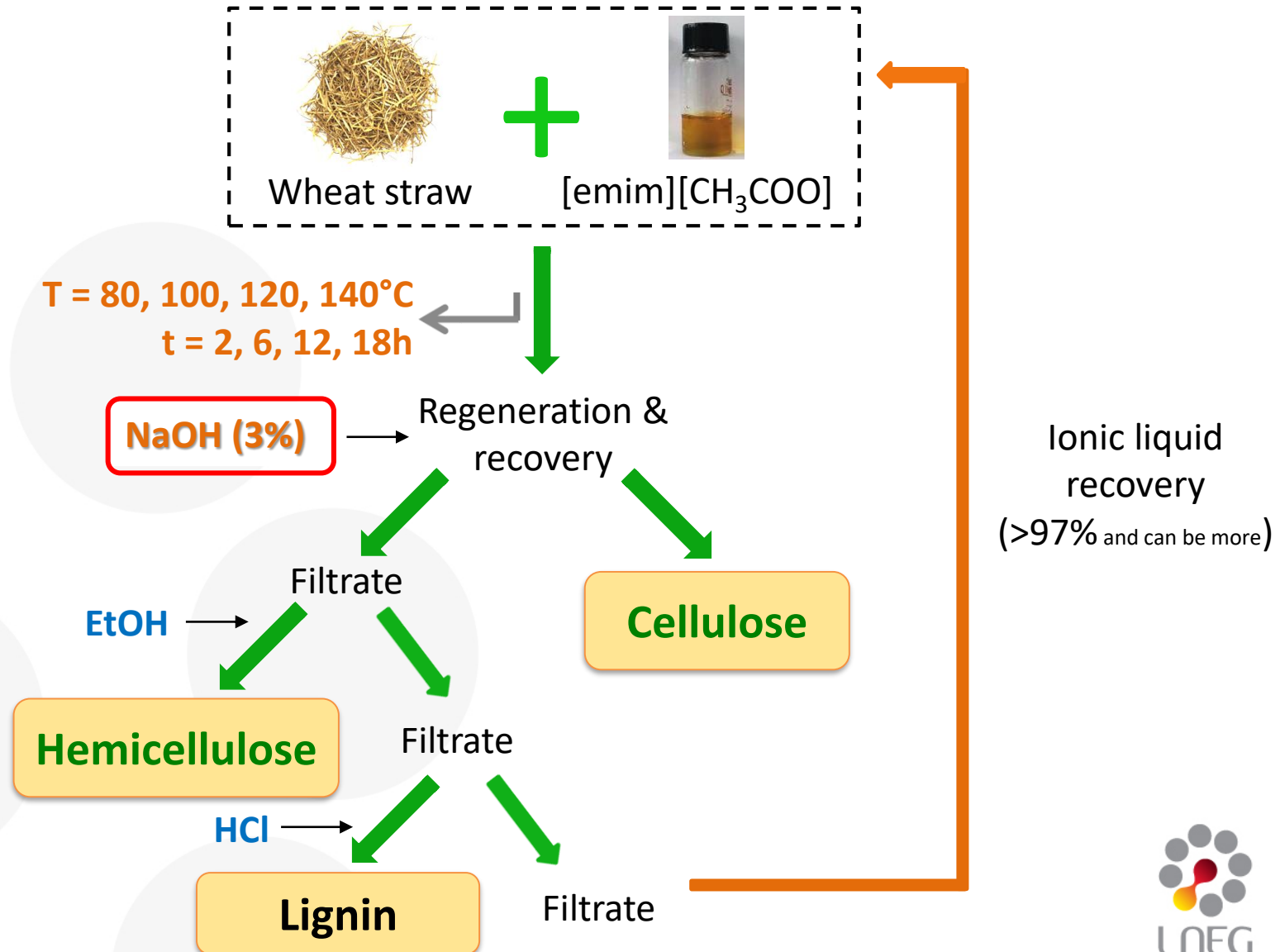
- 1) Alter the physicochemical properties of the biomass macromolecular components;
- 2) Extract a specific macromolecular fraction;
- 3) Perform different fractionation approaches after dissolution.

## Advantages:

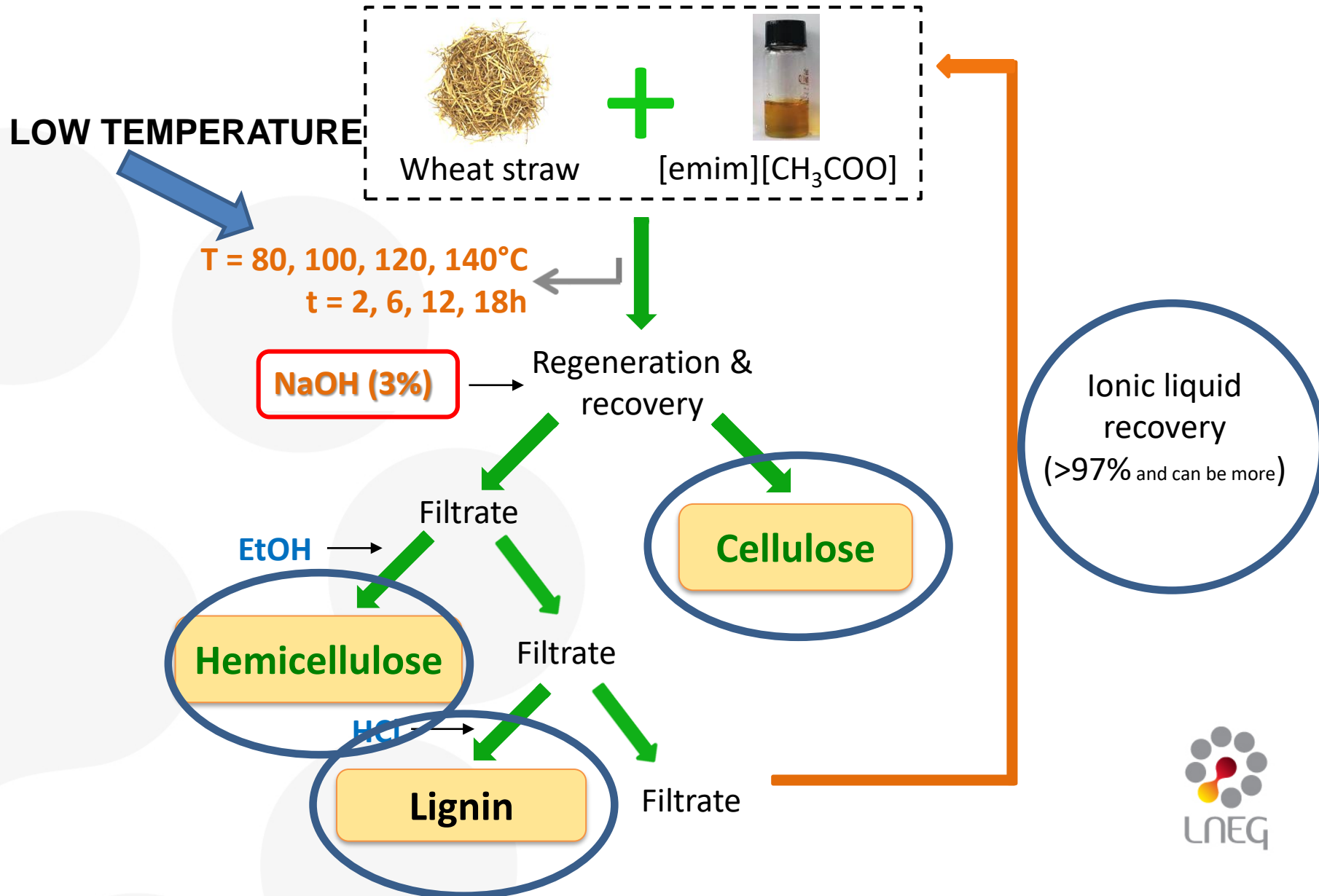
- ✓ ↓ Cellulose crystallinity;
- ✓ ↑ Extraction of lignin
- ✓ Less degradation of monosaccharides;
- ✓ Recyclability and reuse of ILs.



# 3-step biomass fractionation with ILs



# 3-step biomass fractionation with ILs



## Novel pre-treatment and fractionation method for lignocellulosic biomass using ionic liquids

Cite this: *RSC Advances*, 2013, 3, 16040

Sara P. Magalhães da Silva,<sup>ab</sup> André M. da Costa Lopes,<sup>a</sup> Luisa B. Roseiro<sup>a</sup> and Rafał Bogel-Lukasik<sup>\*a</sup>

*Bioresource Technology* 142 (2013) 198–208



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### Pre-treatment of lignocellulosic biomass using ionic liquids: Wheat straw fractionation



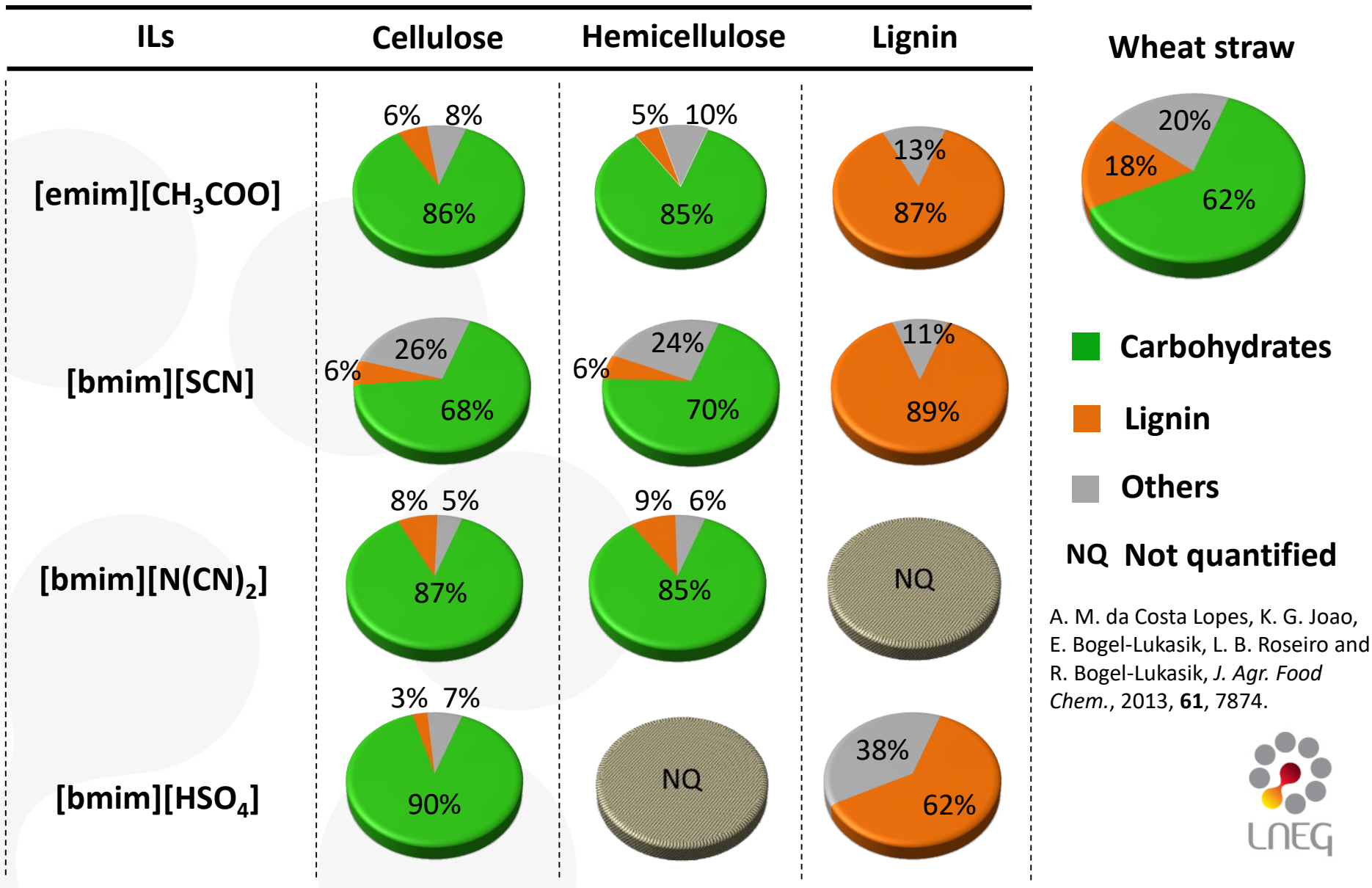
André M. da Costa Lopes<sup>a</sup>, Karen G. João<sup>a</sup>, Djonatam F. Rubik<sup>ab</sup>, Ewa Bogel-Lukasik<sup>c</sup>, Luís C. Duarte<sup>a</sup>, Jürgen Andreas<sup>b</sup>, Rafał Bogel-Lukasik<sup>a,\*</sup>

<sup>a</sup> Laboratório Nacional de Energia e Geologia, Unidade de Bioenergia, 1649-038 Lisboa, Portugal

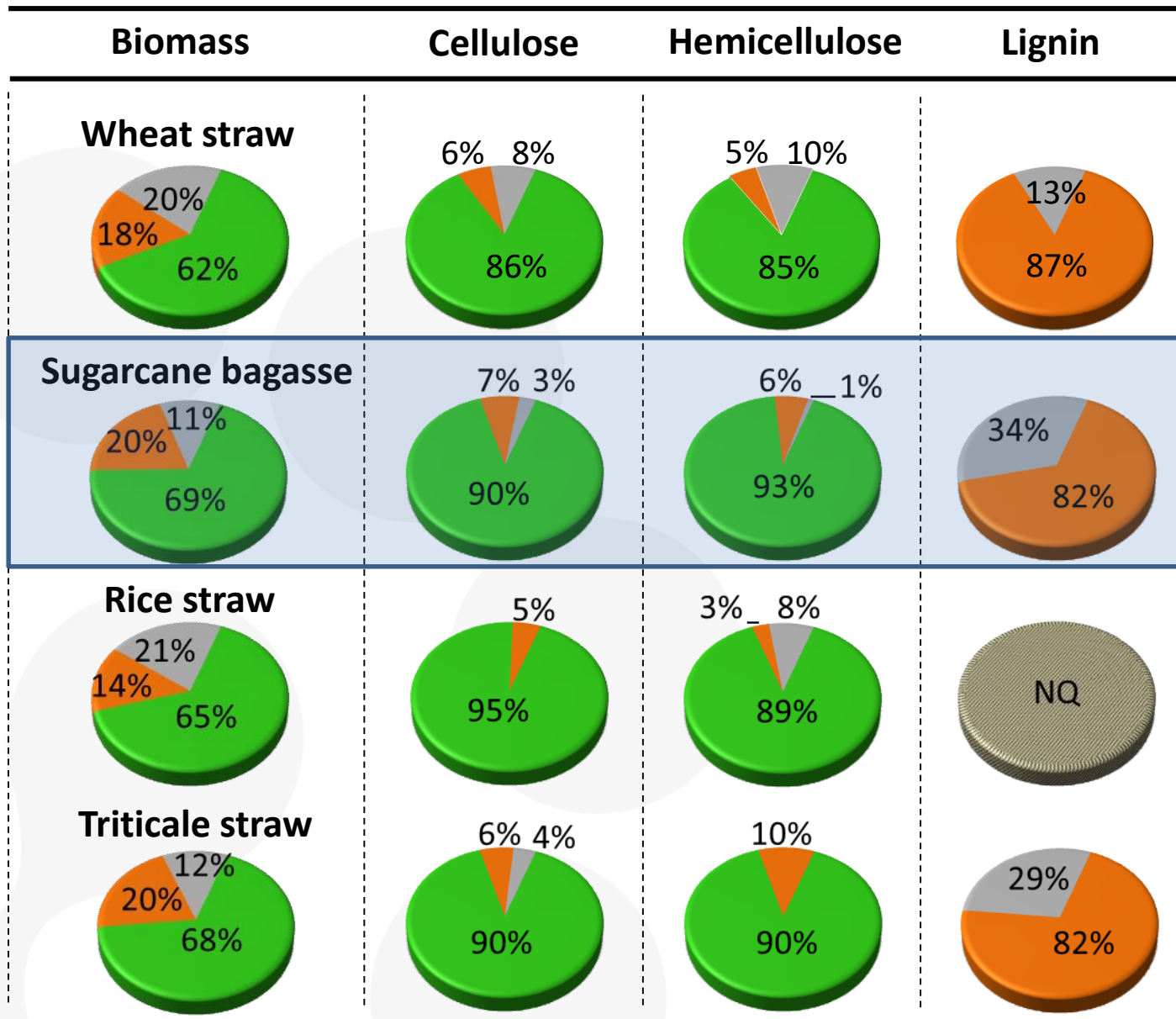
<sup>b</sup> Universidade Regional de Blumenau, Departamento de Química, 89012-900 Blumenau, Brazil

<sup>c</sup> Universidade Nova de Lisboa, Faculdade de Ciências e Tecnologia, Departamento de Química, RRQUNMTE, 2829-516 Caparica, Portugal

# Results with different ILs



# Results with different types of biomass

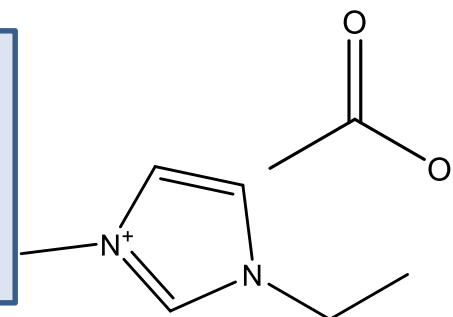


■ Carbohydrates

■ Lignin

■ Others

NQ Not quantified



[emim][CH<sub>3</sub>COO]

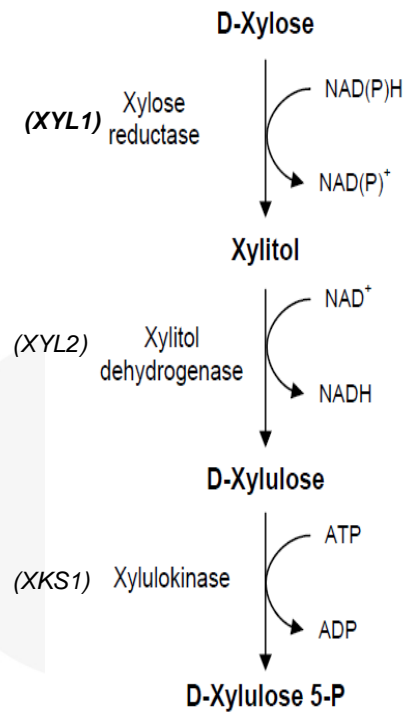
A. M. da Costa Lopes, K. G. Joao, E. Bogel-Lukasik, L. B. Roseiro and R. Bogel-Lukasik, *J. Agr. Food Chem.*, 2013, **61**, 7874.



# New Cell Factories: more efficient



## C5/C6 strain development



LUND UNIVERSITY

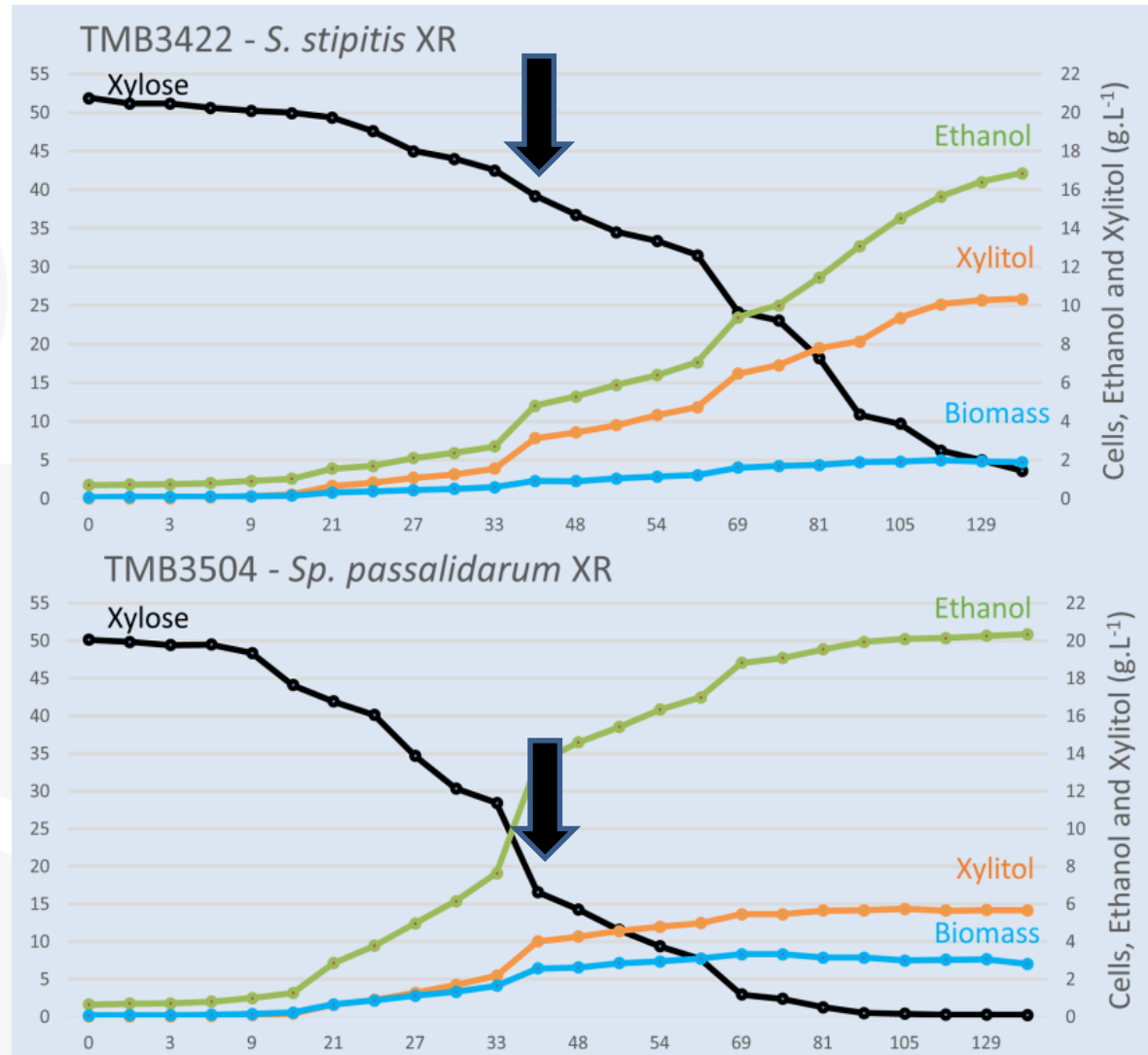
↪ *Spathaspora passalidarum* – 2 *XYL1* genes (xylose reductase)

➤ *XYL1.1* – NADPH dependent

➤ *XYL1.2* – NADPH/NADH

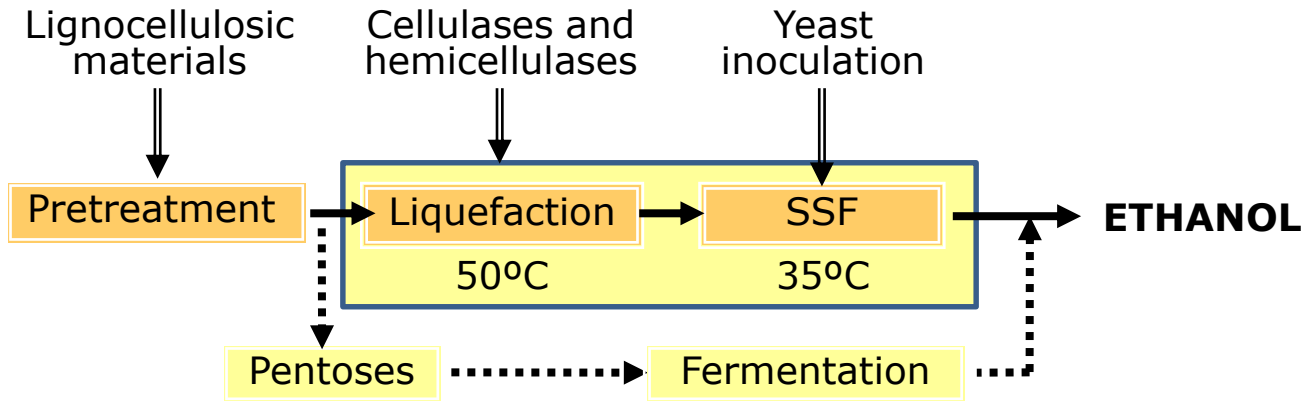
↪ *SpXYL1.2* expressed in *S. cerevisiae* (TMB 3504) – higher affinity for NADH as cofactor – improved xylose fermentation

(Cadete *et al.*, 2016)



# Bioprocess intensification

## Process Intensification: High-solids content ( $\geq 25\%$ DM)

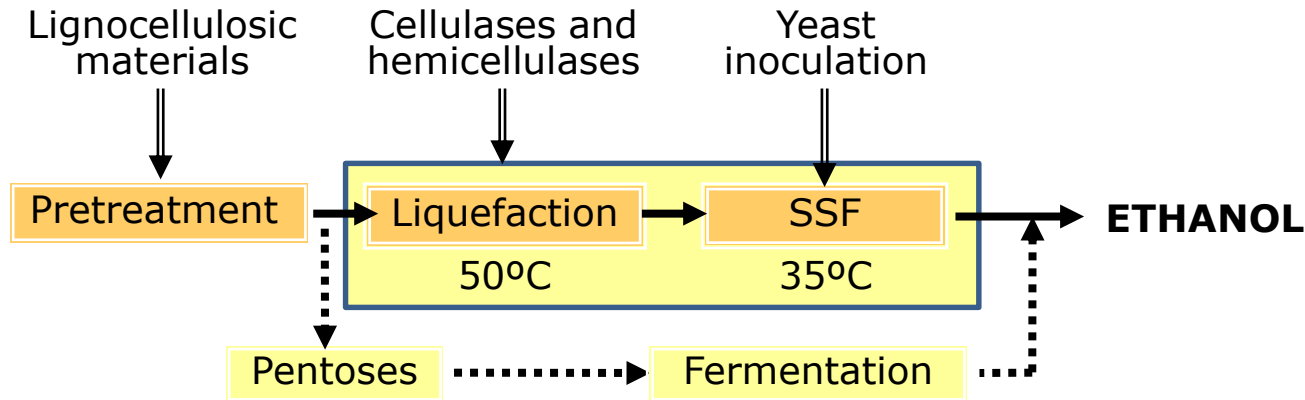


⇒ **Liquefaction step in a rotating drum bioreactor** (24h)

Transfer to 1 L stirred tank reactor, inoculation + **SSF** (48h).



# Bioprocess Intensification



## Sugarcane bagasse High-solids SSF (25-30% DM)

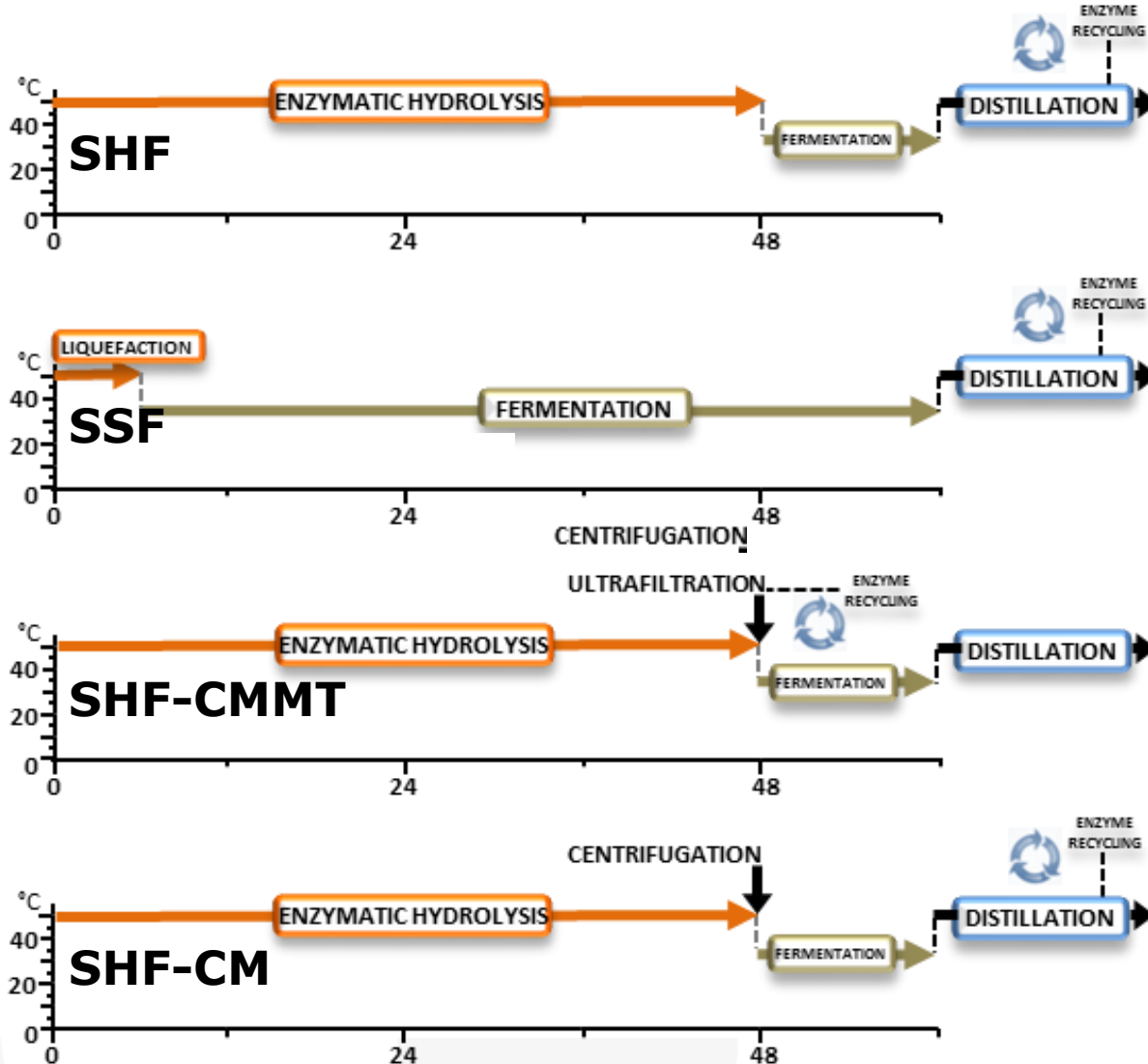
SSF runs at 25 and 30% (w/w) WIS, 10, 20 and 30 FPU/g glucan

Enzyme load (FPU/ g glucan)	% solid content (w/w)					
	25			30		
	Glucose at 24h	Ethanol (g/l)	LSSF yield (%)	Glucose at 24h	Ethanol (g/l)	LSSF yield (%)
10	92	57	52	116	76	53
20	108	72	61	142	87	59
30	127	80	67	157	92	61

# Bioprocess integration



## Comparing process configurations for ethanol yield and enzyme recycling



*Stand-alone technology – 2G*

*Clear mash technology (Brazil) – 1G2G*

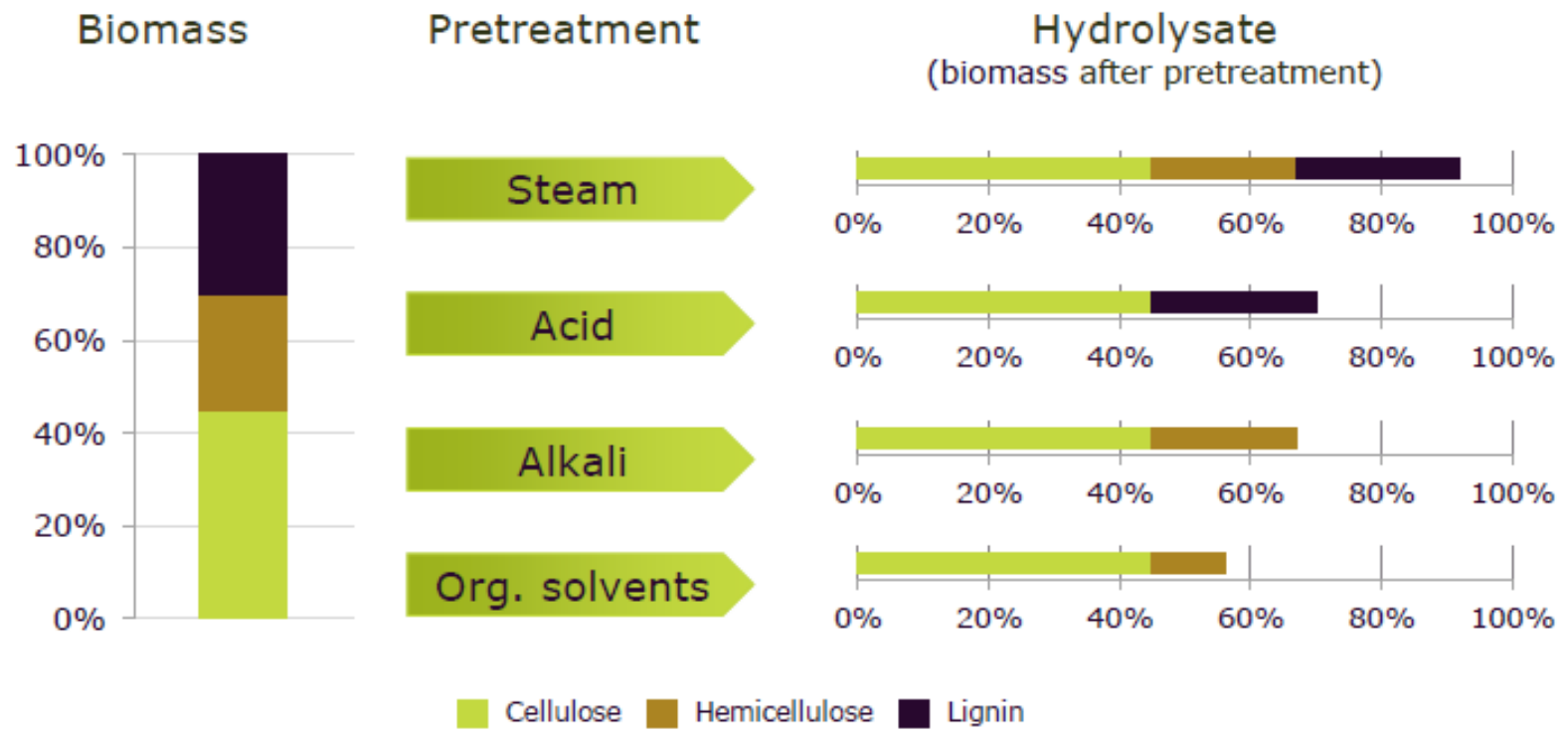


# Enzyme Technology: Moving to tailor-made real cases

AS INDUSTRIALIZED PROCESSES ARE LOCKED INTO PLACE, HIGHLY CUSTOMIZED TECHNOLOGIES ARE NEEDED



- Different pretreatment technologies produce different hydrolysates and inhibitors
- Hemicellulose mix (C5 sugar types) also vary. Enzyme components must be tailored to match

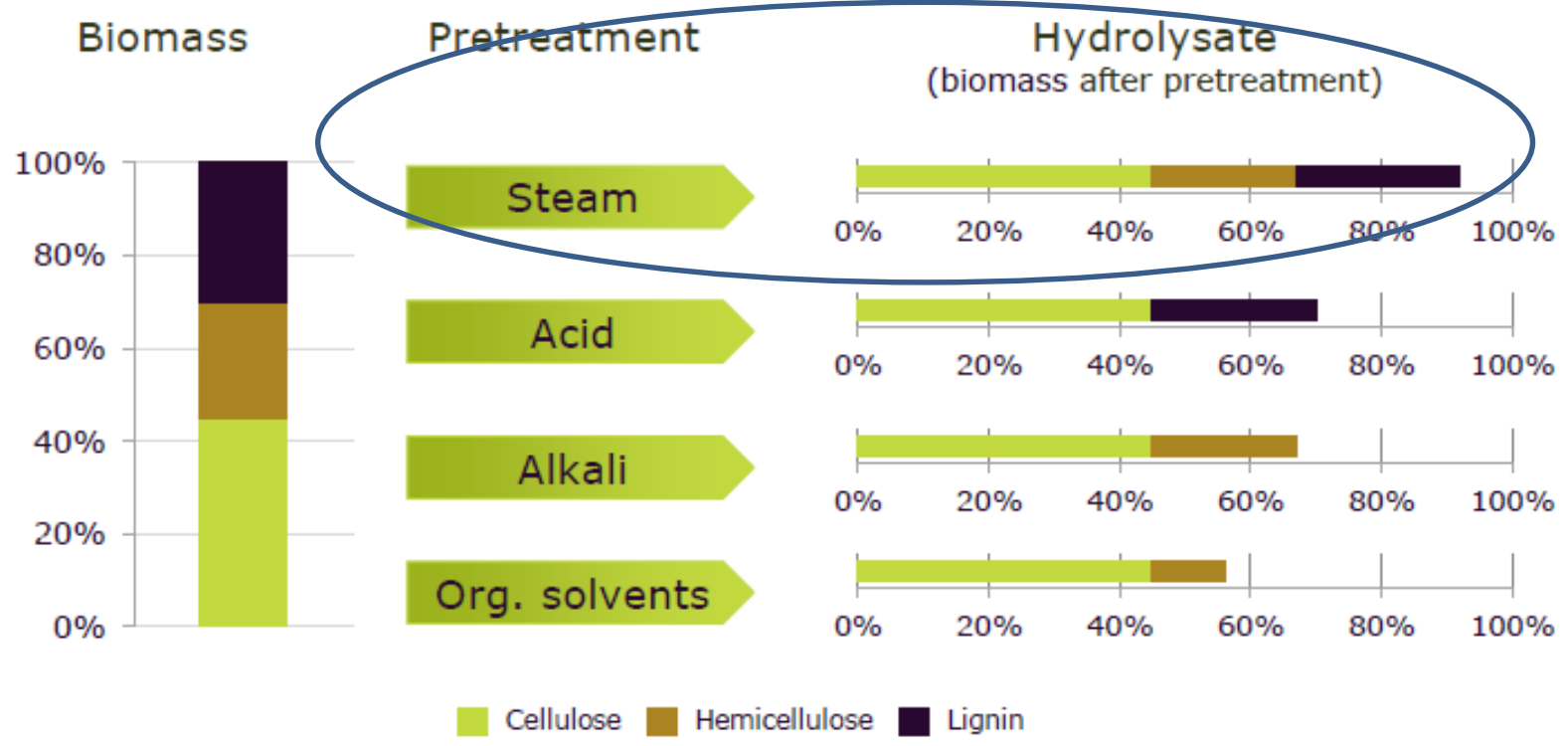


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# What are the next Achievements?

- Is ETOH the right “building block” for NextGen transportation sector?
  - Future role of higher alcohols, long-chain fatty acids,...still uncertain
- Improving overall energy-efficient (eg, cane-energy, low-demand biomass pretreatments, CBP, DSP....)
  - Biochemicals and other chemicals shall have an increasing importance in advanced biorefineries
  - However, there are few chemical products with markets large enough to absorb the production of a large-scale biofuel biorefinery
  - Is lignin becoming the “gold component” as main feedstock for conversion into high-added value products, being EtOH production a co-product of the value chain? (e.g., BALI™ from Borregard Industries)
- Do we need ex-situ EH (adding external cellulases and other hydrolytic enzymes) in the future?
- Clusters-based biorefineries shall use more efficient the entire feedstocks and by-streams (CAPEX & OPEX also decreases) and it is expected as industrial outcome a wider range of products for different “core” markets.



# Questions?

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