Confidential

Presentations Sarpsborg, Norway
11 May 2010

Part 1. Development of integrated lignocellulose biorefinery for co-production of chemicals, transportation fuels, electricity and heat
Overview & results of the IP BIOSYNERGY (FP6)

**Hans Reith, René van Ree, Reyes Capote Campos,
Robert Bakker, Paul de Wild, Fréderic Monot, Boris Estrine,
Tony Bridgwater, Alessandro Agostini**

and

Part 2. Thermolysis of lignin for value-added products; Progress catalytic lignin pyrolysis at ECN

Paul de Wild and Hans Reith

ECN-L--10-073 JULY 2010

Part 1 Development of integrated lignocellulose biorefinery for co-production of chemicals, transportation fuels, electricity and heat; Overview & results of the IP BIOSYNERGY (FP6)




Development of integrated lignocellulose biorefinery for co-production of chemicals, transportation fuels, electricity and heat

Overview & results of the IP BIOSYNERGY (FP6)

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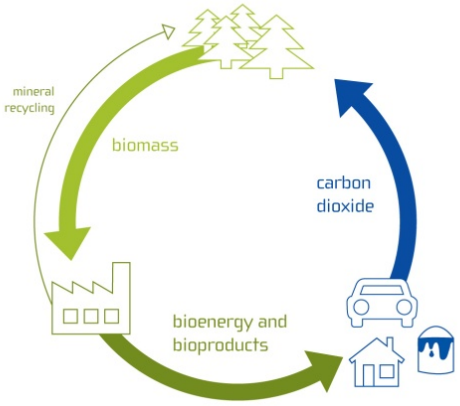
11th May 2010, Sarpsborg, Norway

Contents

- Background of the IP BIOSYNERGY
- Overview and highlights technology development and design activities
- Preliminary conclusions and perspectives



Features Integrated Project BIOSYNERGY

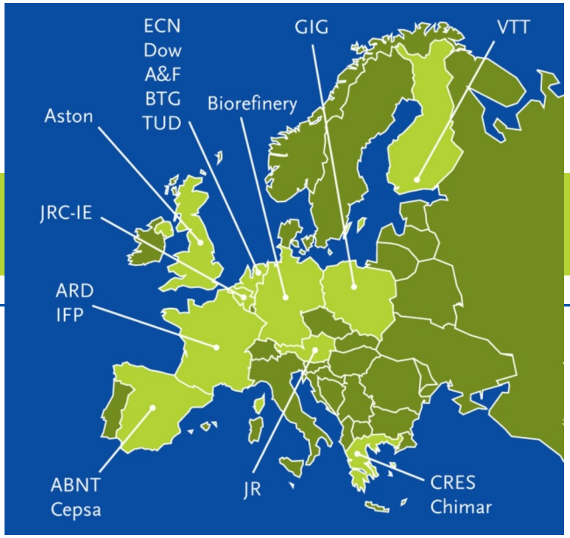
BIOmass for the market competitive and environmentally friendly **SYN**thesis of bio-products – chemicals and/or materials – together with the production of secondary en**ERGY** carriers – transportation fuels, power and/or CHP – through the biorefinery approach.

- Overall aim: Development **multiproduct cellulose-ethanol based biorefinery** technology
- **Focus on valorisation of residues from cellulose ethanol production** to make the production of this biofuel more cost competitive
- Bioprocessing and thermochemical pathways combined
- Process development from lab-scale to demonstration at pilot-scale.

EU FP6 Program: Contract No. 038994 – SES 6. EC Officer: Silvia Ferratini.
Duration: 1-1-2007 – 31-12-2010 (48 months). Budget: 13.4 M€, EC grant 7M€

BIOSYNERGY

Consortium

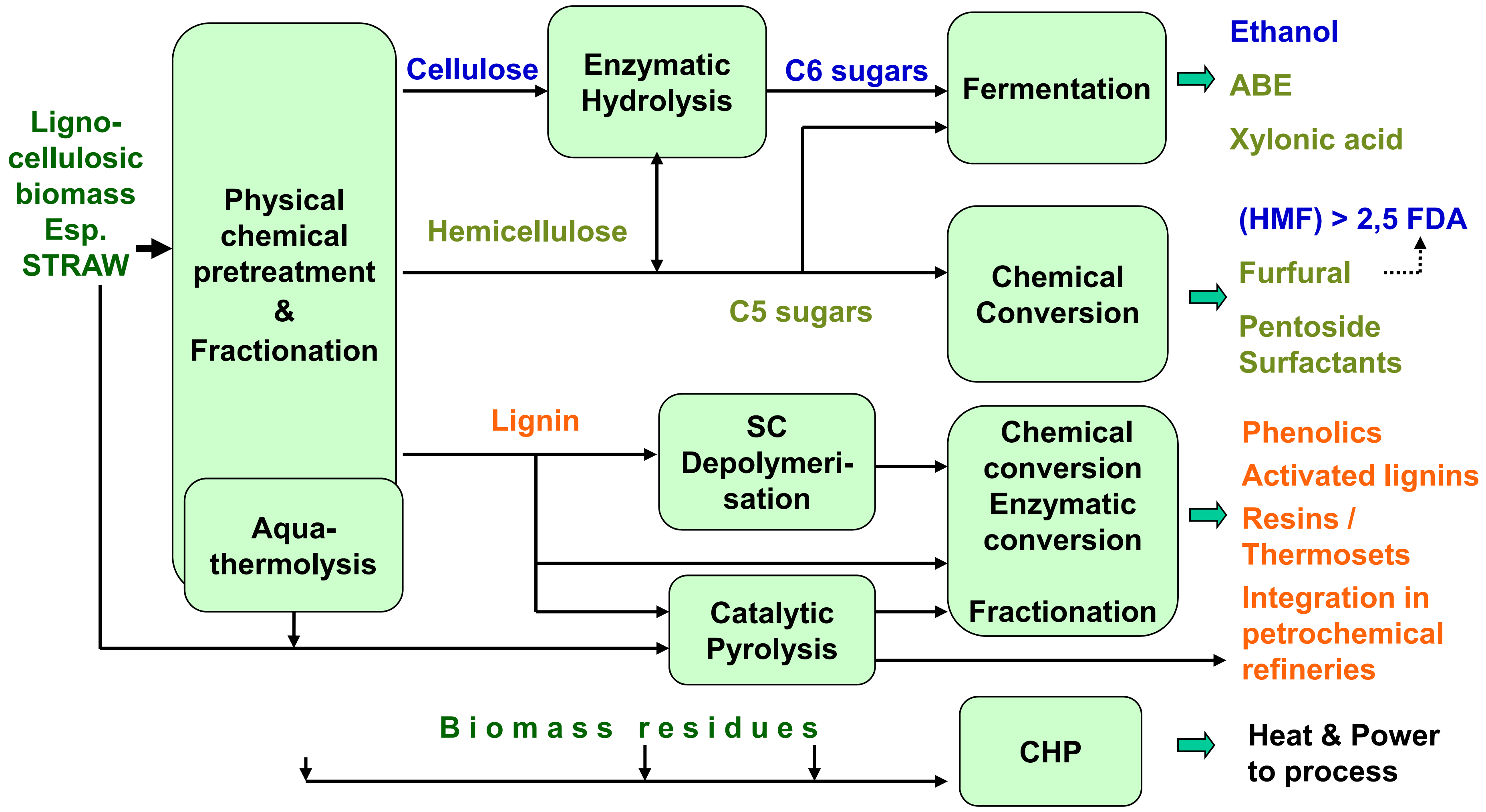


17 partners from industry (7), R&D institutes (8) and Universities (2) from 10 EU countries

1	Energy research Centre of the Netherlands (ECN)	The Netherlands	NL
2	Abengoa Bioenergía Nuevas Tecnologías S.A. (ABNT)	Spain	ES
3	Compania Espanola de Petroles S.A. (Cepsa)	Spain	ES
4	DOW Benelux B.V. (Dow)	The Netherlands	NL
5	VTT Technical Research Centre of Finland (VTT)	Finland	FI
6	Aston University (Aston)	United Kingdom	UK
7	WUR Agrotechnology and Food Innovations B.V. (A&F)	The Netherlands	NL
8	Agro Industrie Recherches et Développements (ARD)	France	FR
9	Institut Francais du Pétrole (IFP)	France	FR
10	Centre for Renewable Energy Sources (CRES)	Greece	EL
11	Biomass Technology Group (BTG)	The Netherlands	NL
12	Joanneum Research Forschungsgesellschaft m.b.H. (JR)	Austria	AT
13	Biorefinery.de (Biorefinery)	Germany	DE
14	Glowny Instytut Gornictwa (GIG)	Poland	PL
15	Joint Research Centre – Institute for Energy (JRC-IE)	The Netherlands	NL
16	Chimar Hellas S.A. (Chimar)	Greece	EL
17	Delft University of Technology (TUD)	The Netherlands	NL

Product lines in the IP BIOSYNERGY

Multi-product biorefinery, Focus on residues cellulose ethanol: **C5 and lignin valorisation**

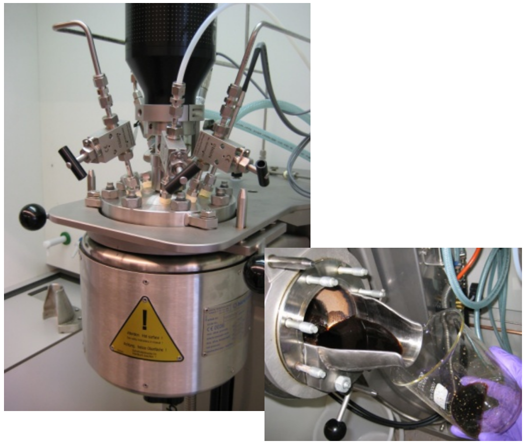


Advanced physical/chemical fractionation (WP1)

- Major cost factor in biorefinery
- Model feedstocks: **wheat straw**, woods

Processes studied

- Mechanical/Alkaline Fractionation (MAF; A&F)
- Ethanol/water Organosolv (ECN)
- Organic acid organosolv (Avidel process; ARD)
- Acid hydrolysis (Biorefinery.de)
- Reference technology: steam explosion (ABNT)



Ethanol/H_2O Organosolv,ECN

Mech/alk pretreatment A&F

Acid organosolv Pilot plant ARD

Partners: A&F, ABNT, ARD, Bioref, ECN, TUD

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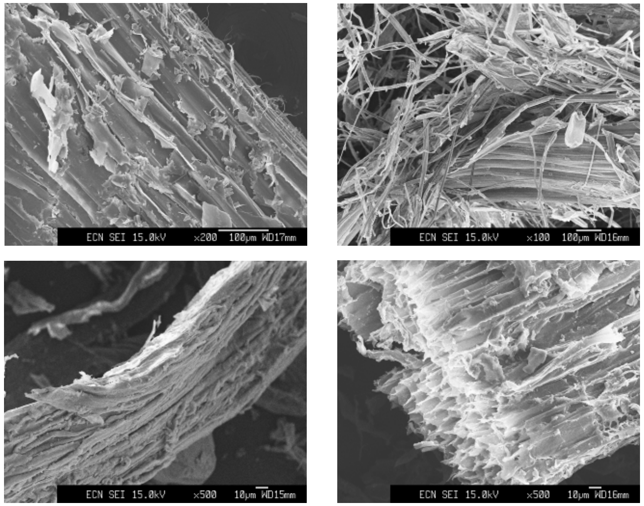
Ethanol/water organosolv: efficiency of pretreatment and lignin quality

- Good pre-treatment hardwoods and esp. straw
- Effective extraction of hemicellulose and lignin
- Cellulose fibrous structure remains intact
- Good access for enzymes: Hydrolysis yield > 90%
- Purity and MW distribution of lignins indicate suitability for chemicals production
- Solvent recycle crucial for energy use and costs

Fresh Pre-treated

Wheat straw

Willow



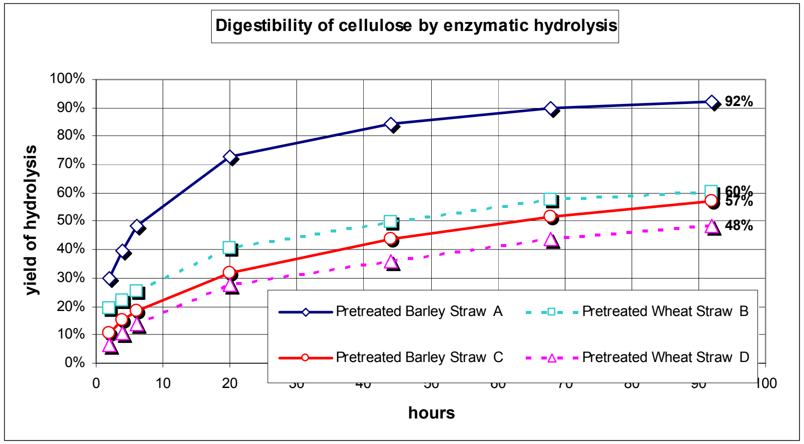
200°C, 60 min, EtOH-H_2O 60:40 (w/w)

Biomass	Pulp yield (dw%)	Xylan hydrolysis (%)	Delignification (%)	Enzymatic degradability (% cellulose feedstock)
Barley straw	51	80	57	92
Wheat straw	62	45	55	60
Willow	66	50	64	71
Poplar	71	28	ND	39
Spruce	73	NA	33	ND

Feedstock / sample	Mw	PD
Poplar	2419	3.5
Barley straw	3006	4.1
Willow	3452	4.1
Alcell®	2985	3.6

Conclusions pretreatment/fractionation

- No clear “winner”: All studied routes lead to significant fractionation of C5, C6 sugars and lignin from lignocellulose
- Differences in cellulose hydrolysis yields



- Processes need to be optimised toward a particular goal, for example:
  - Hemicellulose hydrolysis for further processing of C5
  - High enzymatic degradability of the cellulose fraction
  - Recovery of a high quality lignin stream (**>>organosolv , MAF**)
- Review and results benchmark to be published

Lignin products from Organosolv Fractionation (ECN)

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Innovative thermo-chemical conversion (WP2)

Focus on thermochemical processing of **biomass and lignin.**

Development of:

- Hybrid, staged processing concept: aquathermolysis followed by fast pyrolysis
- Catalytic fast pyrolysis *(in 2nd part)*
- Separation and upgrading technology

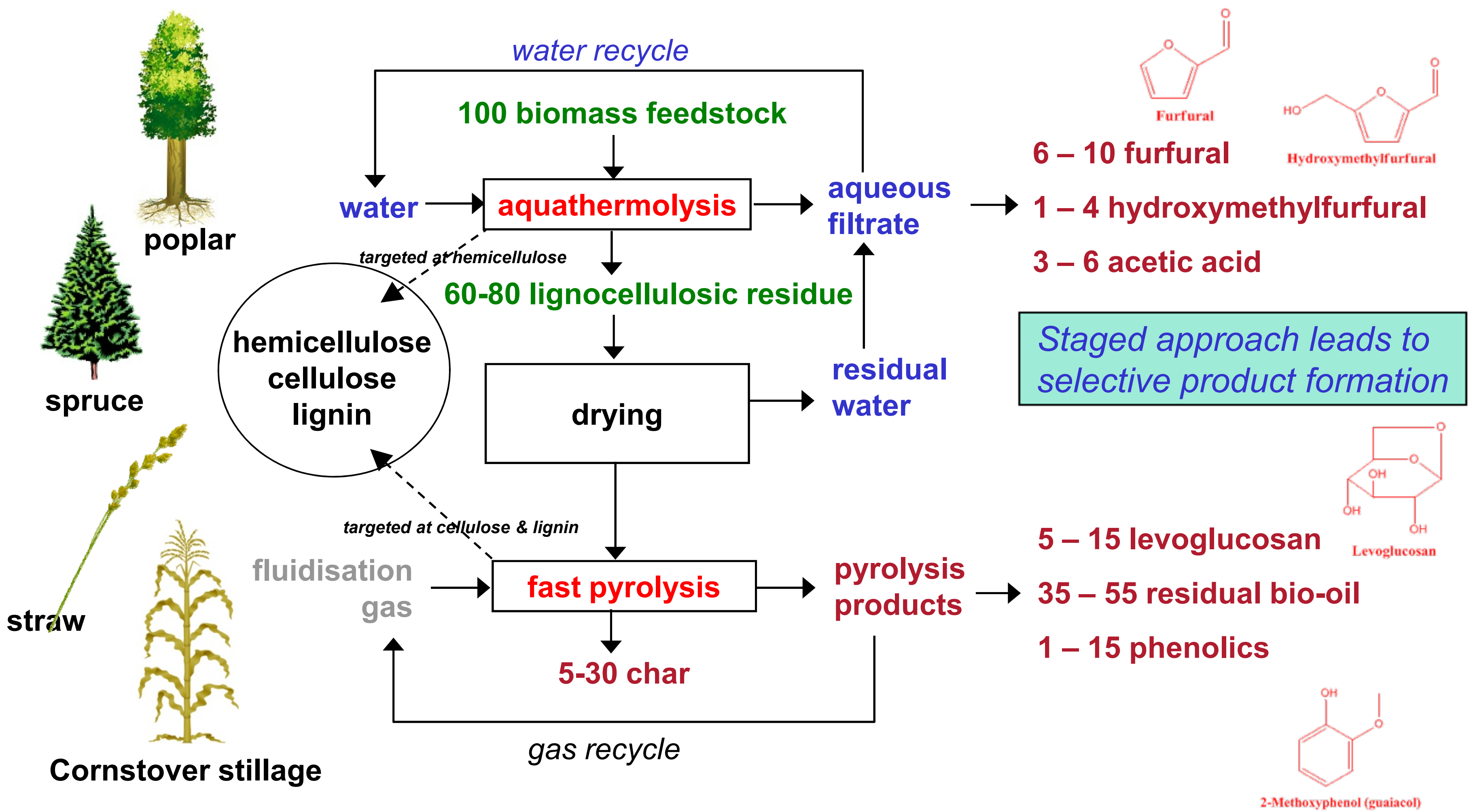
Partners: ECN, Aston, BTG

BFB reactor ECN

BIOSYNERGY Innovative thermo-chemical conversion (WP2)

Hybrid staged process for biomass conversion

- aquathermolysis: pressurised hot water (pre)treatment (200 °C, 30 min.)
- followed by BFB fast pyrolysis (350-400°C, vapour residence time 1-2 sec.)

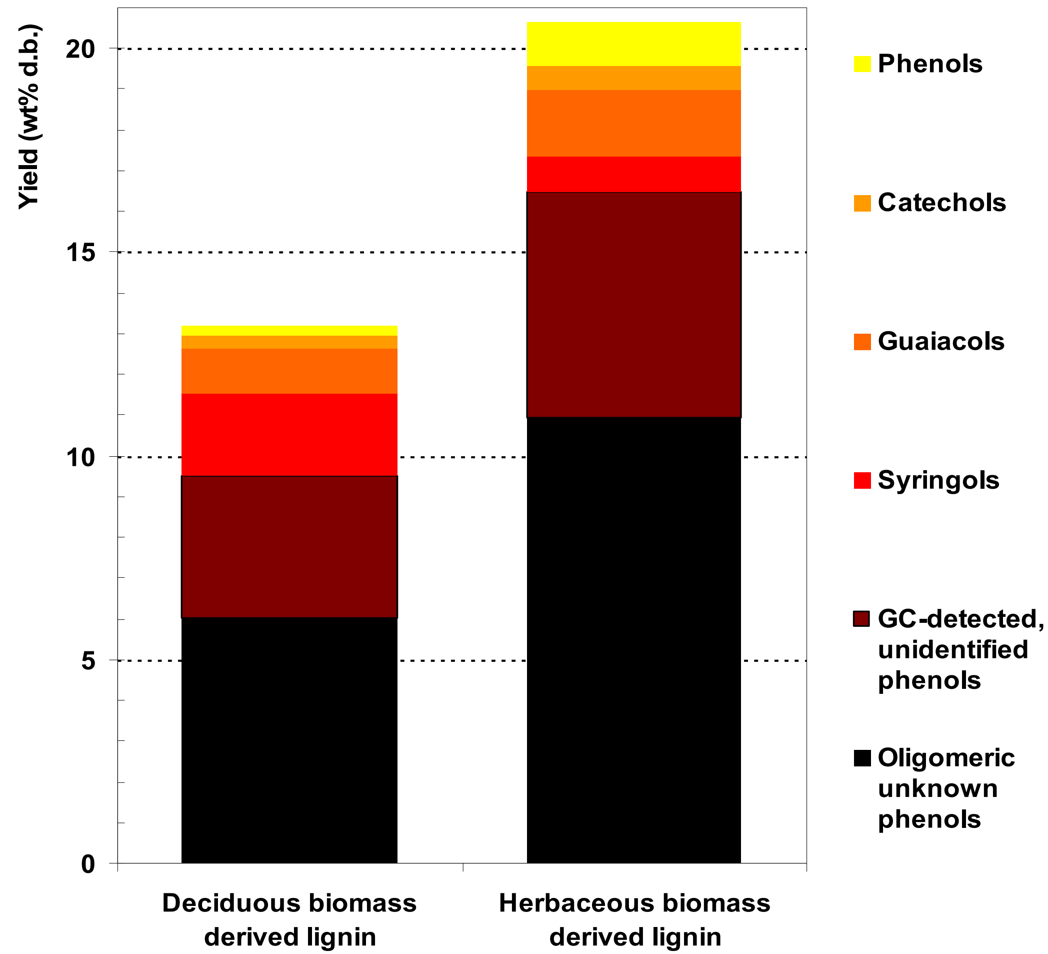


Continuous catalytic pyrolysis of lignin: results

100 lignin pyrolyse to:
- 15 - 20 gas (CO, CO_2, CH_4)
- 20 - 25 water
- **15 - 25 organic condensables**
- 30 - 35 solid (char)

continuous cat. pyrolysis at 400°C gives **high yields of phenolics:**

- **13 wt% (d.b.)** phenolics from hard wood derived Alcell organosolv lignin
- **20 wt% (d.b.) phenolics** from herbaceous derived lignin from soda pulping of grass/straw mixture
- apparently the herbaceous lignin is easier to crack



BIOSYNERGY

Upgrading of bio-oils

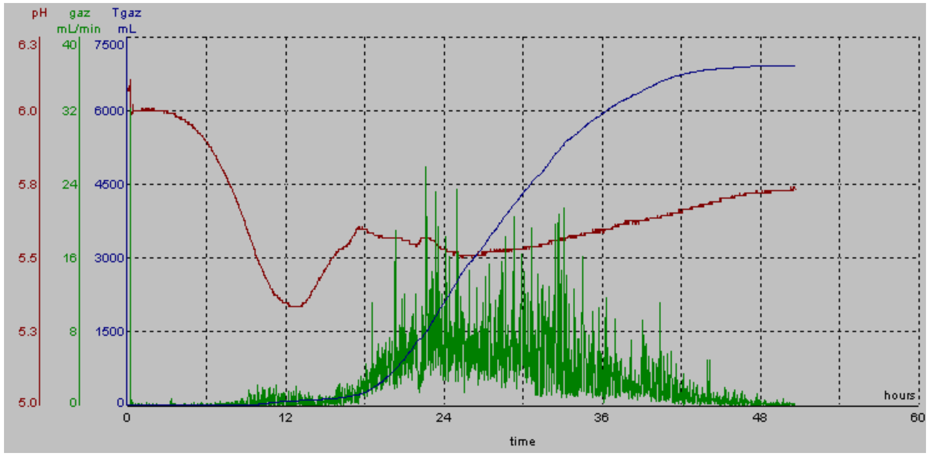
Successful development of:

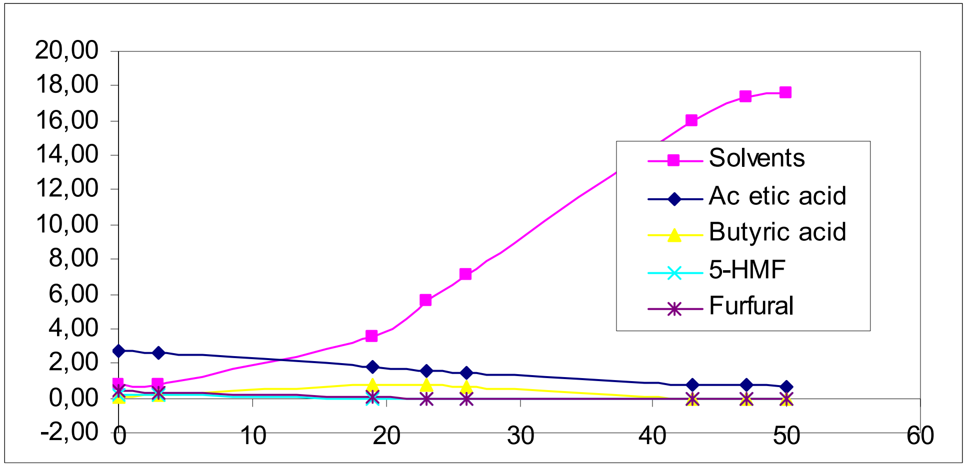
- Procedures to improve quality of fast pyrolysis oil (filtration, dewatering)
- Fractionation of bio-oil into enriched fractions suitable for resins and wood preservatives

80-250 kg/hr rotating cone fast pyrolysis pilot plant at BTG

ABE Fermentation wheat straw hemicellulose hydrolyzate IFP/A&F

- Successful screening and selection of strains on pure substrates
- **ABE Production on wheat straw hemicellulose hydrolyzates prepared by steam explosion** in mild acidic conditions
  - 50% Hydrolysate in synthetic medium (60 g/L total sugars (Glu 9; Xyl 51 g/L)
  - Strain *Clostridium beijerinckii* NCIB 8052 / pH controlled at 5.3
- **Good lab scale results : Final solvents (ABE) : 17,6 g/L**
- **Continuous fermentation still a challenge**
- **ABE separation from fermentation broth rotating disc separator demonstrated**





Xylonic acid fermentation and lignin activation (VTT)

Xylonic acid production from xylose

- Successful fermentation results in batch and continuous cultures
- acid hydrolyzed DDGS and wheat straw pentose hydrolysates

Functional lignin derivatives: lignin 'activation'

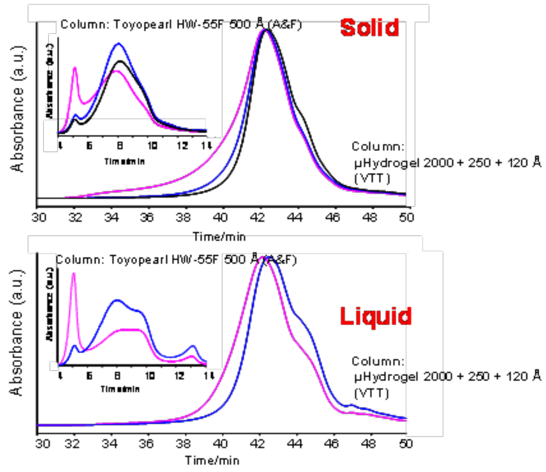
- *Aim:* improvement of reactivity (cross linking behaviour) to enhance product options
- Successful enzymatic lignin modification by *Trametes hirsuta* laccases

Characterization of modified lignin polymers by chemical and spectroscopic methods.

ThL treated lignin

Solubilized / Control lignin

Raw lignin / unsolubilised / untreated lignin



Mattinen et al. (2008). "Polymerization of different lignins by laccase," *BioResources* 3(2), 549-565.
Mattinen et al, (2009) "Modifications of lignans by Trametes hirsuta laccase" *BioResources* 4(2), 482-496.

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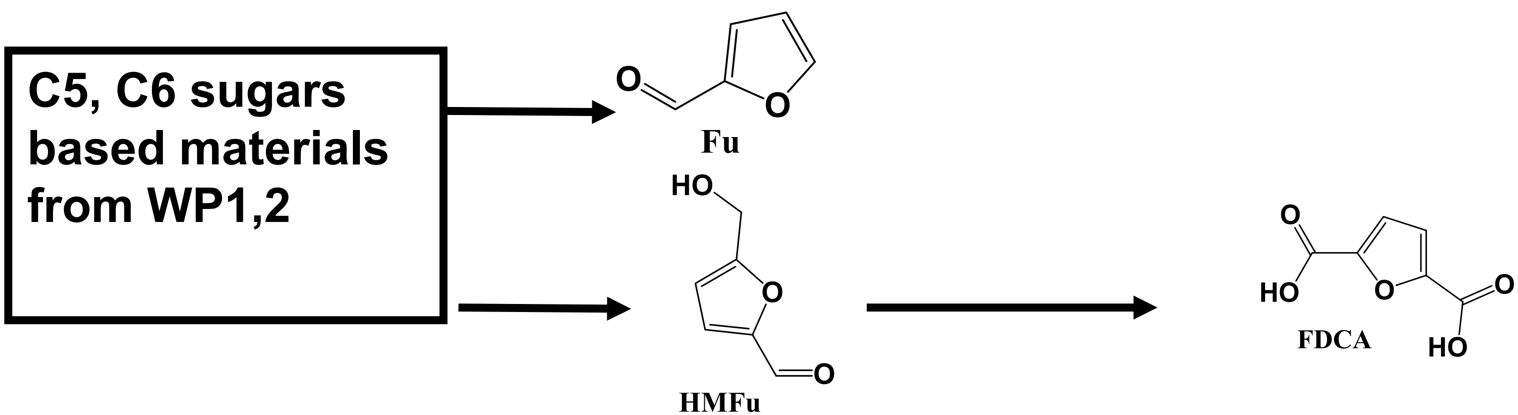
Production & characterisation platform chemicals (WP4)

Partners: DOW, A&F, ARD, Bioref, GIG, Chimar, TUD

- Lignin depolymerisation in supercritical CO2 (A&F) **Improved yields >10%**
- Analysis kinetics furfural (Fu) synthesis from xylose and modelling to improve furfural production process: TUDelft. **Yield improvement > 85%**



- Hydroxymethylfurfural production from glucose dehydration>> **substantial yield improvement by use of ionic liquids (Bioref)**
- **Synthesis 2,5-Furandicarboxylic acid (2,5-FDCA)** from HMF with **high yield > 90%** (Bioref/ A&F). FDCA potential replacement for TFA in PET.

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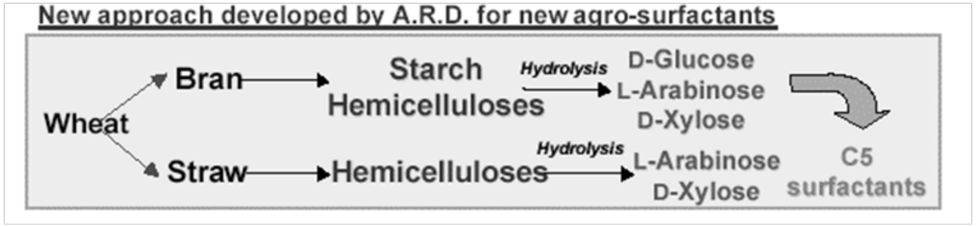
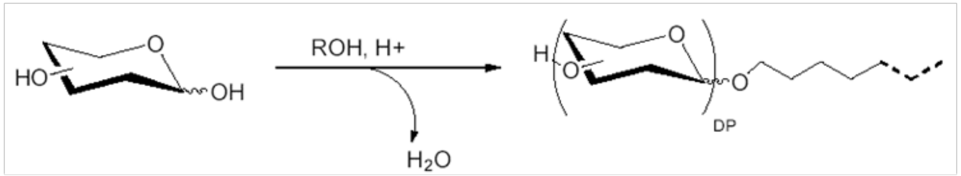
Application testing and market validation

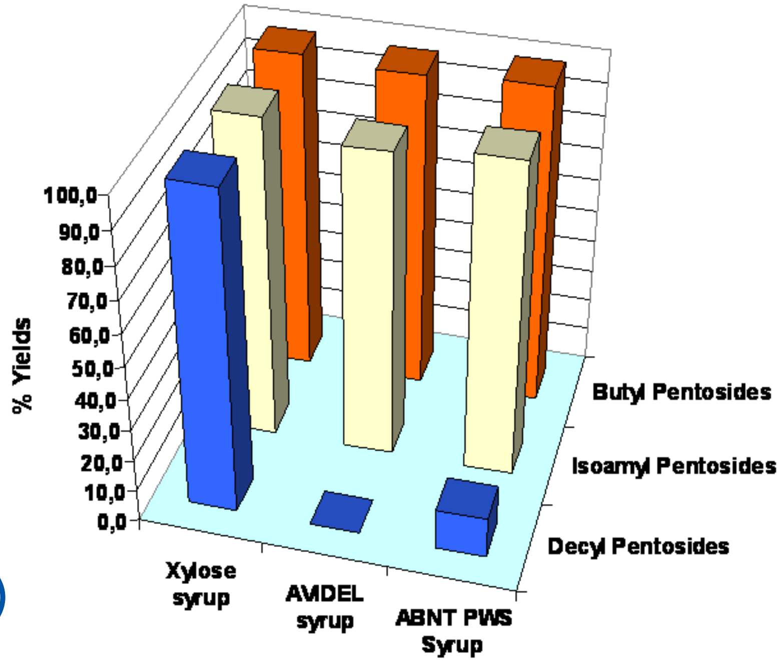
- Application testing 2,5 FDCA-derived polymers: **promising initial results, further work in progress**
- **Phenol substitution up to 25 wt%** by (organosolv) lignin in thermosetting phenol-formaldehyde resins for particle board application (test results Chimar)
- **Performance pentose based surfactants for paper impregnation** comparable to petrochemical products

Pentose valorisation as raw material for surfactants; ARD





- Pentoses + Fatty alcohols (ROH C:4-C:18)
- **Aim**: Production of "green" pentoside surfactants at the price level of petrochemical competitors (~1,500 €/ton)
- Short tail Pentoside surfactants prepared **in quantitative yields from straw derived <u>unpurified</u> pentose syrups**



Yields of Alkyl pentosides obtained for three pentose syrups and for 3 types of alcohols.

Integration of results in Conceptual design biorefinery plant (WP5)

Basic design for integral lignocellulose biorefinery plant at an existing cellulose ethanol site: AB BCyL demonstration plant, Salamanca.

- Targeted outputs:
  - bio-ethanol,
  - chemicals, materials, CHP
- 5 EtOH based biorefinery types
- design in final stage

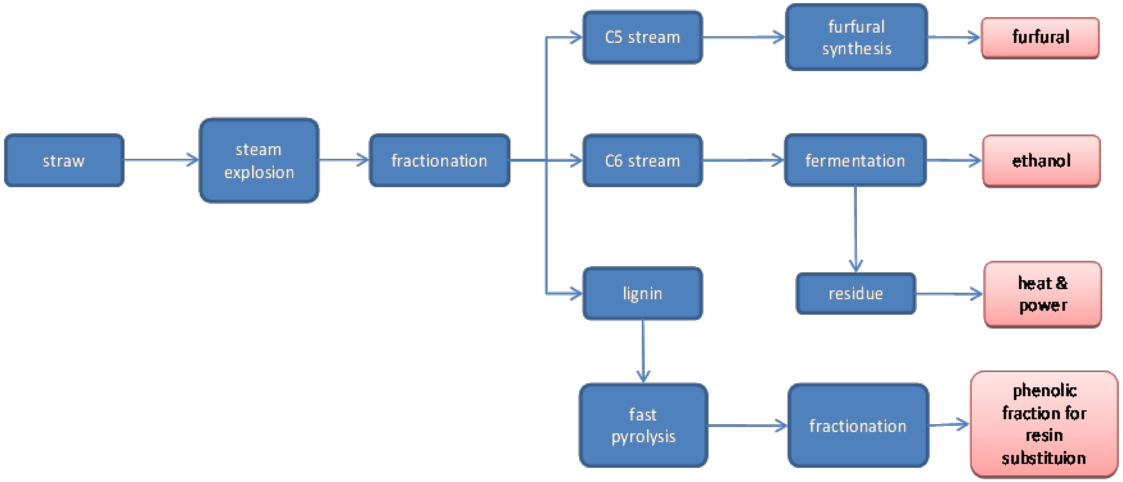
Partners: ABNT, Aston, ECN

*BCyL cellulose ethanol demo plant AB, Salamanca, 5 Million L EtOH/yr from 25,000 ton straw. **Operational since Oct. 2009**.*

Biomass-to-products chain design (WP6, Aston)

Aim: Identification of the most promising biorefinery chains for the EU

- **Modelling tool with modular structure** developed
- **Process synthesis and simulation**: ongoing, in final stage
- **Process comparison** using **MCDA** (**incl. economics, LCA, socio-economics**)



Focus on ethanol based biorefineries: Biorefinery co-producing **ethanol, furfural, phenolic resins and CHP**

*Preliminary results economic evaluation indicate that **biorefinery processes** (ethanol + chemicals) have **better economic perspective** than cellulose ethanol only plus lignin combustion for steam generation.*

Partners: **Aston**, ECN, IFP, CRES, JR, JRC, Cepsa, ABNT.

Preliminary conclusions & perspectives

- Biosynergy RTD provides a **solid basis for valorization of C5 sugars and lignin**, fitting in a cellulose ethanol based biorefinery concept. Substantial advances reached on lab/bench scale. Several processes will be demonstrated on pilot scale in 2010.

- **Pretreatment technologies** need to be optimised toward a particular goal/end products. This requires **Integrated development** of the trajectory Feedstock<>pretreatment<>hydrolysis<>fermentation.

- **Lignin valorization to chemicals** is an important tool for **economic profitability** of the biorefinery. **Promising results** attained for:
  - direct application of (organosolv) lignin in resins: 25 wt% phenol substitution
  - catalytic pyrolysis of lignin to phenolics
  - enzymatic lignin conversion (laccases) to improve reactivity

- **Biorefinery of lignocellulose to ethanol + chemicals + CHP shows better economic perspectives than production of only cellulose ethanol + CHP.**

- Development of integrated processes / chain optimisation is a major success factor AND one of the major challenges.

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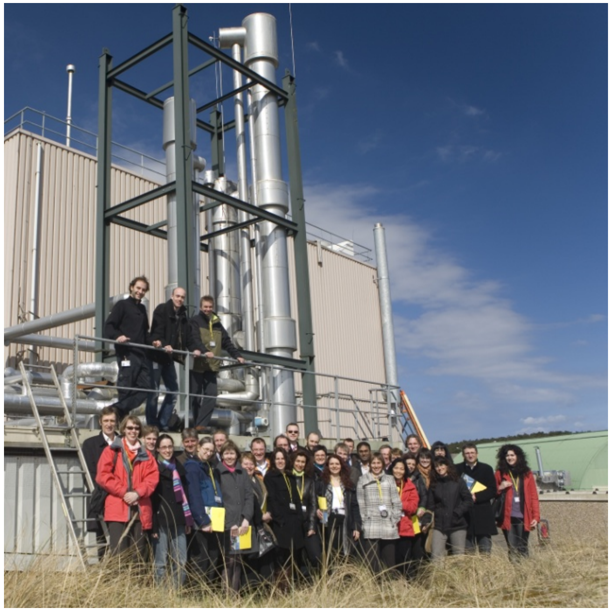

BIOSYNERGY Team during Technical Excursion at ECN in April 2008. Photo: Jasper Lensselink.

BIOSYNERGY

Thank you for your attention!

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SIXTH FRAMEWORK PROGRAMME

The Integrated Project BIOSYNERGY is supported by the European Communities through the Sixth Framework Programme for Research and Technological Development (2002–2006) with a grant up to 7.0 million € under contract number 038994 – (SES6). The project duration is 1-1-2007 – 31-12-2010 (48 months).

EC Officer: Silvia Ferratini.

Part 2 Thermolysis of lignin for value-added products; Progress catalytic lignin pyrolysis at ECN

ECN

Energy research Centre of the Netherlands

Thermolysis of lignin for value-added products

Progress catalytic lignin pyrolysis at ECN

Paul de Wild and Hans Reith

11 May 2010, Sarpsborg, Norway

Energy research Centre of the Netherlands ECN

- 600 highly qualified employees
- Annual turnover: ca. 100 million Euro
- 10 -15 international patents granted each year
- Co-operation with companies, universities and research institutes

Mission: ECN develops and brings to market high-quality knowledge and technology for a sustainable energy system

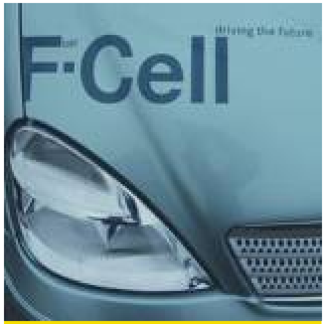
ECN: Trias energetica

Solar Energy

Biomass

Wind Energy

Efficiency & Infrastructure



H_2 & Clean Fossil Fuels

Policy Studies

Engineering & Services

Unit Biomass, Coal & Environmental Research: 60 f.t.e.

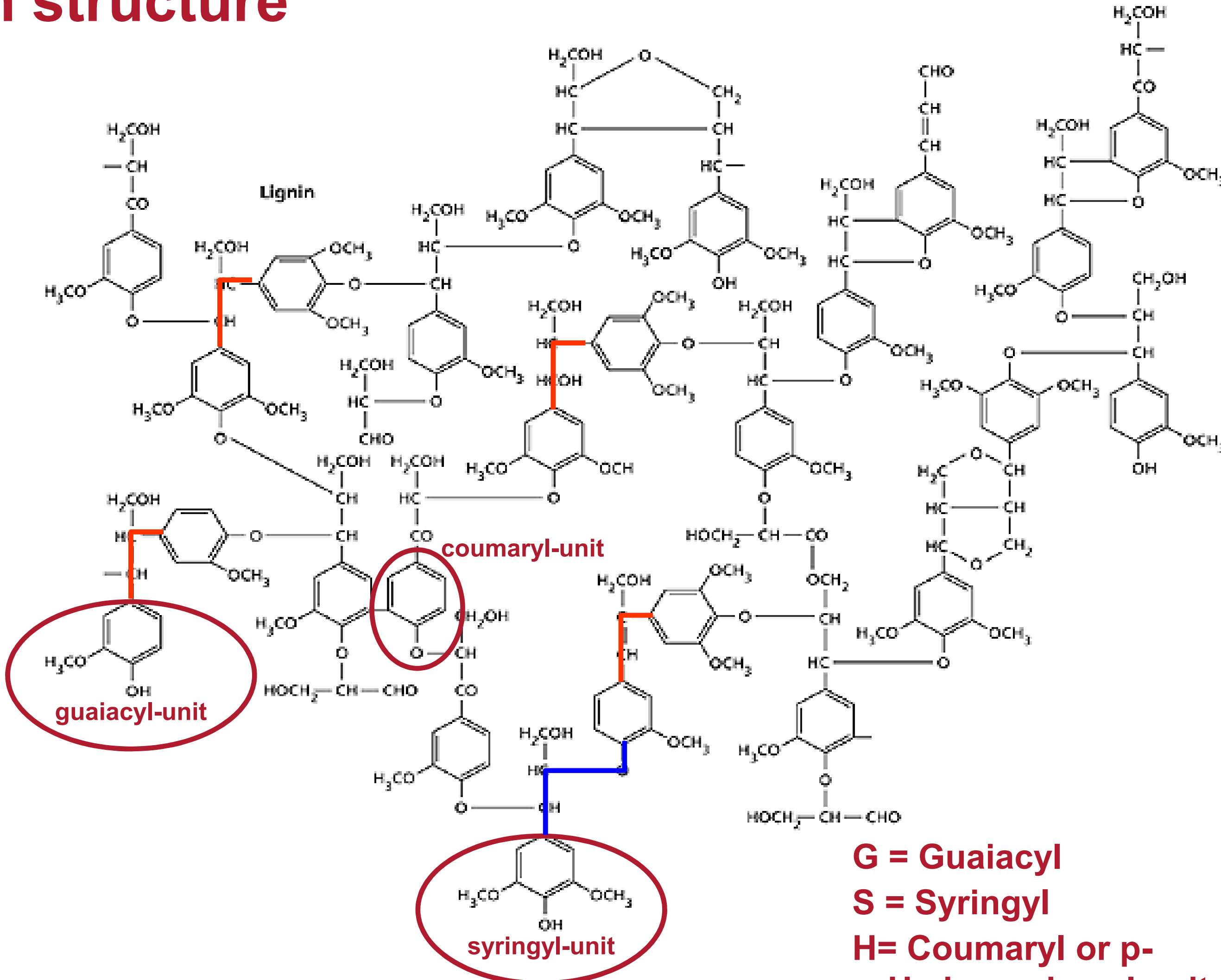
- Biomass and coal co-firing; biomass torrefaction
- Gasification: Substitute Natural Gas (SNG), Syngas
- **Biofuels and chemicals: pre-treatment, pyrolysis, catalytic biomass conversion, process design, LCA**
- Emission reduction technology
- Environmental research

Outline

- Background of Lignin valorisation
- Experimental
- Results
- Conclusions

Simplified lignin structure

- Heterogeneous and recalcitrant structure
- Most bonds are of the **β-O-4** type
- Most internal bonds via the **para** position
- Great variety of other bonds
- Guaiacyl, syringyl and coumaryl (p-Hydroxyphenyl) units are the predominant structural units
- Structure is an idealised formula of a deciduous lignin



G = Guaiacyl
S = Syringyl
H= Coumaryl or p-Hydroxyphenyl units

Main components lignocellulosic biomass

Large variation in composition per taxonomic group

Deciduous woods
(e.g. beech, poplar,..)

40-50% cellulose
30-40% hemicellulose

20-25% lignin
(syringyl & guaiacyl units)

Coniferous woods
(e.g. spruce, pine,…)

40-45% cellulose
25-30% hemicellulose

25-30% lignin
(mainly guaiacyl units)

Herbaceous crops
(e.g. wheat, grass, corn,…)

40-45% cellulose
35-45% hemicellulose

15-25% lignin
(p-hydroxyphenyl, guaiacyl & syringyl units)

Lignin valorisation

- Lignin is worlds' second most abundant natural polymer containing valuable aromatic (phenolic) structures
- Lignin itself is a valuable resource for performance products
- Lignin is the main constituent of large residual streams in e.g. the pulp and paper sector and (future) cellulose EtOH plants, biorefineries,…

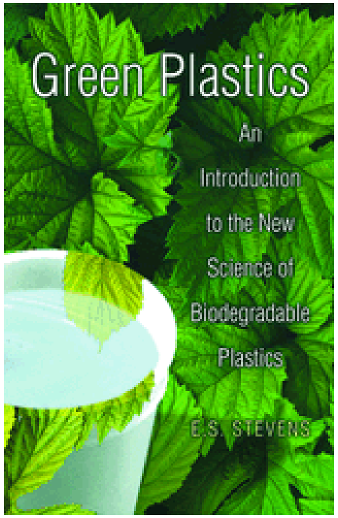
Technologies

- Combustion for heat and/or power (main application to date)
- Gasification for syngas
- Hydroliquefaction for transportation fuels (reformulated gasoline)
- Direct application 'organosolv' lignins in resins*
- Enzymatic processing (laccases) to improve reactivity*
- **Pyrolysis for chemicals, performance products and fuels***

Successful valorisation of lignin is a key-issue for an economically viable lignocellulosic biorefinery.

Potential applications lignin derived phenolics

Adhesives, resins

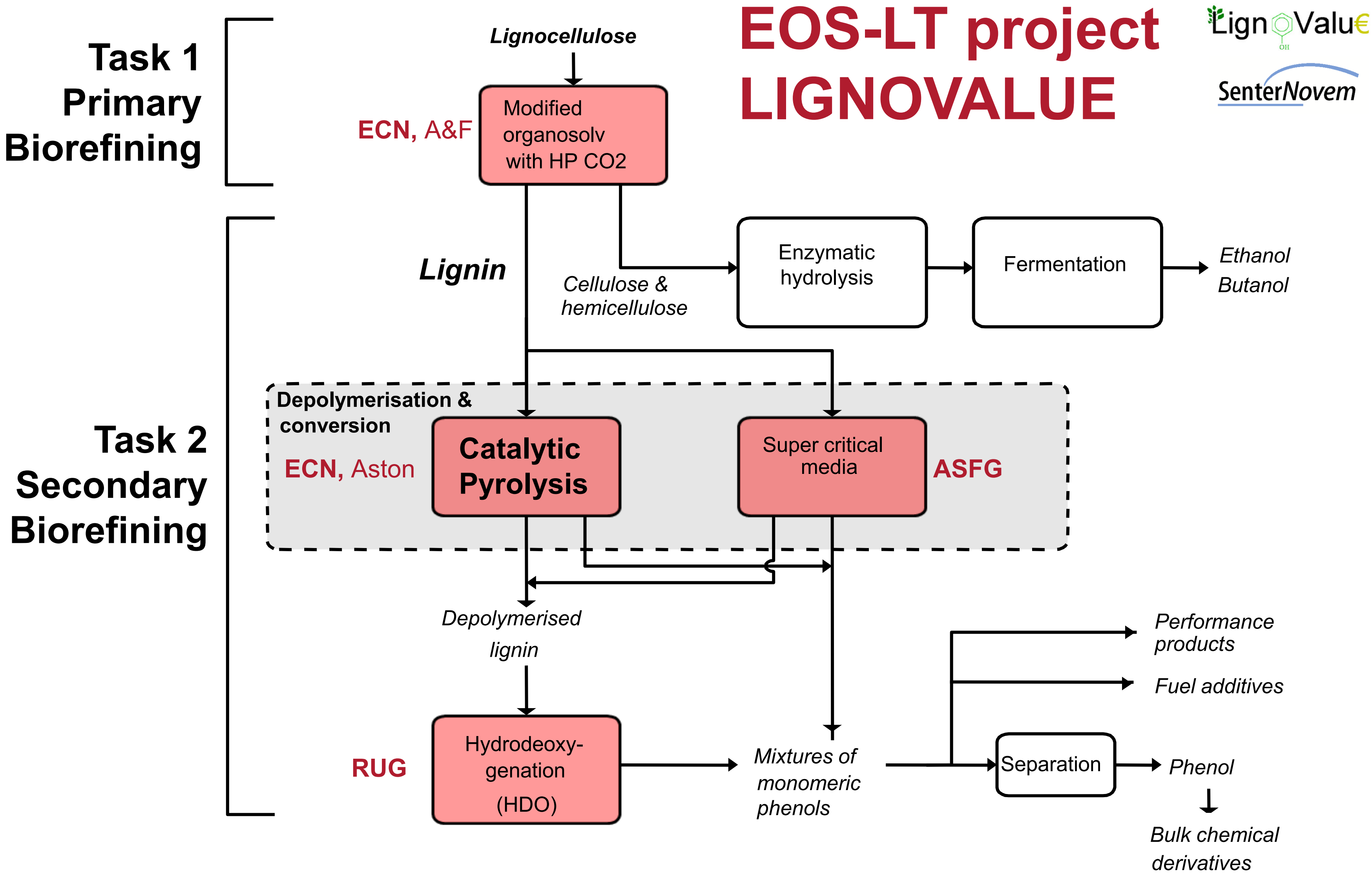


Bio-based plastics
- epoxies
- polyolefins

- **Fuel additives (e.g. aromatic ethers, cyclohexanone)**
- **BTX**
- **Binders**
- **Bio-bitumen**

Specialty phenolics for high-value applications such as fragrances and pharmaceuticals

EOS-LT project LIGNOVALUE



Lignin feedstocks used for pyrolysis

- ‘Technical’ lignins, organosolv, high purity

Winter wheat straw,
(ECN organosolv pulping)

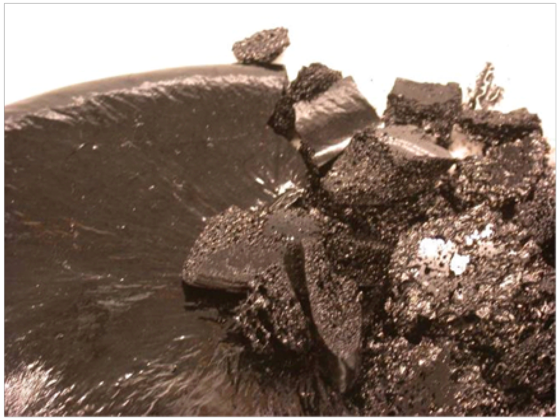
Mix of Sarkanda grass and wheat straw,
(soda pulping, Protobind™)

Mix of hardwoods.
(organosolv pulping, Alcell™)

- Residual lignins (‘stillage’) from cellulose ethanol production

Experimental

- **Feedstock**
  - Pelletised Alcell organosolv lignin from hardwoods
  - Thermal characterisation by fusion tests, thermogravimetry (TGA) and differential scanning calorimetry (DSC)
  - Characterisation by ^{13}C–Solid State–Cross Polarization / Magic Angle Spinning Nuclear Magnetic Resonance (13C-SS-CP/MAS/NMR)



Crushed solidified lignin melt
(melt, cool, crush)

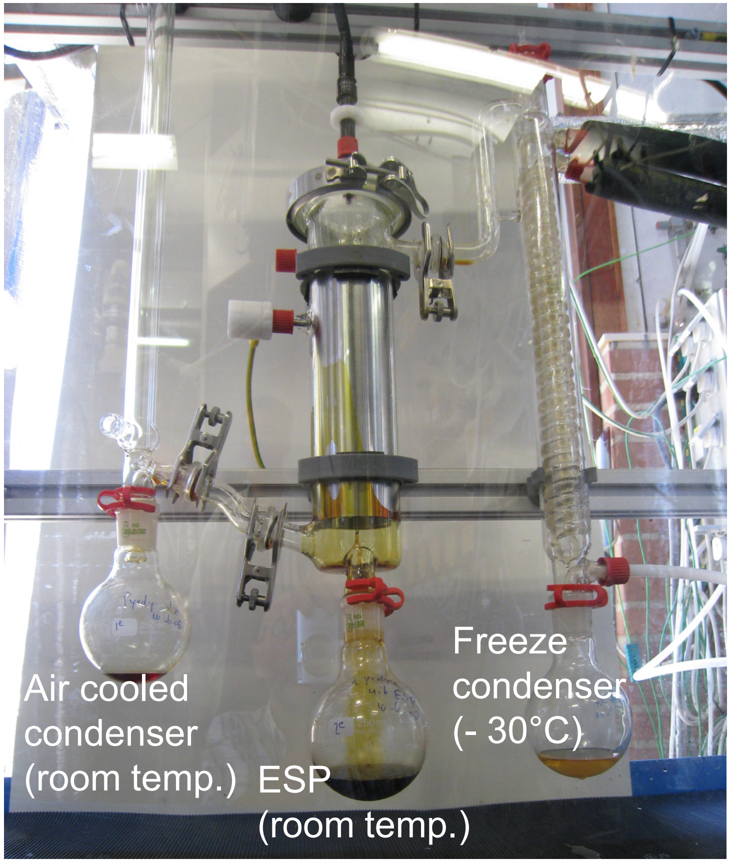
(Alternative: dissolve in EtOH, dry, crush)

- **Experimental conditions pyrolysis tests**
  - Lab-scale atmospheric pressure bubbling fluidised bed (1 kg/hr, 5 kW_{th})
  - Fractionated sampling of products

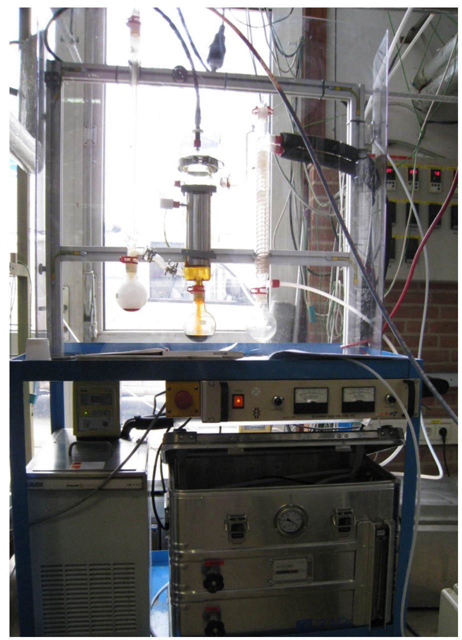
Bubbling fluidised-bed pyrolysis and product collection

- Hot sand-bed, 400-500°C, fluidised with Ar
- Vapour residence times: seconds solid residence time: minutes
- Product collection by CEN/TS tar sampling protocol, using **washing bottles (batch)**. In **continuous experiments sampling by cooled condensers, ESP, cold trap**
- On-line analysis of non-condensable gases
- Off-line analysis of condensable products by GC/MS/FID

PYPO: Pyrolysis Product Obtention



PYPO in action, trapping the products from the pyrolysis of Alcell lignin…..

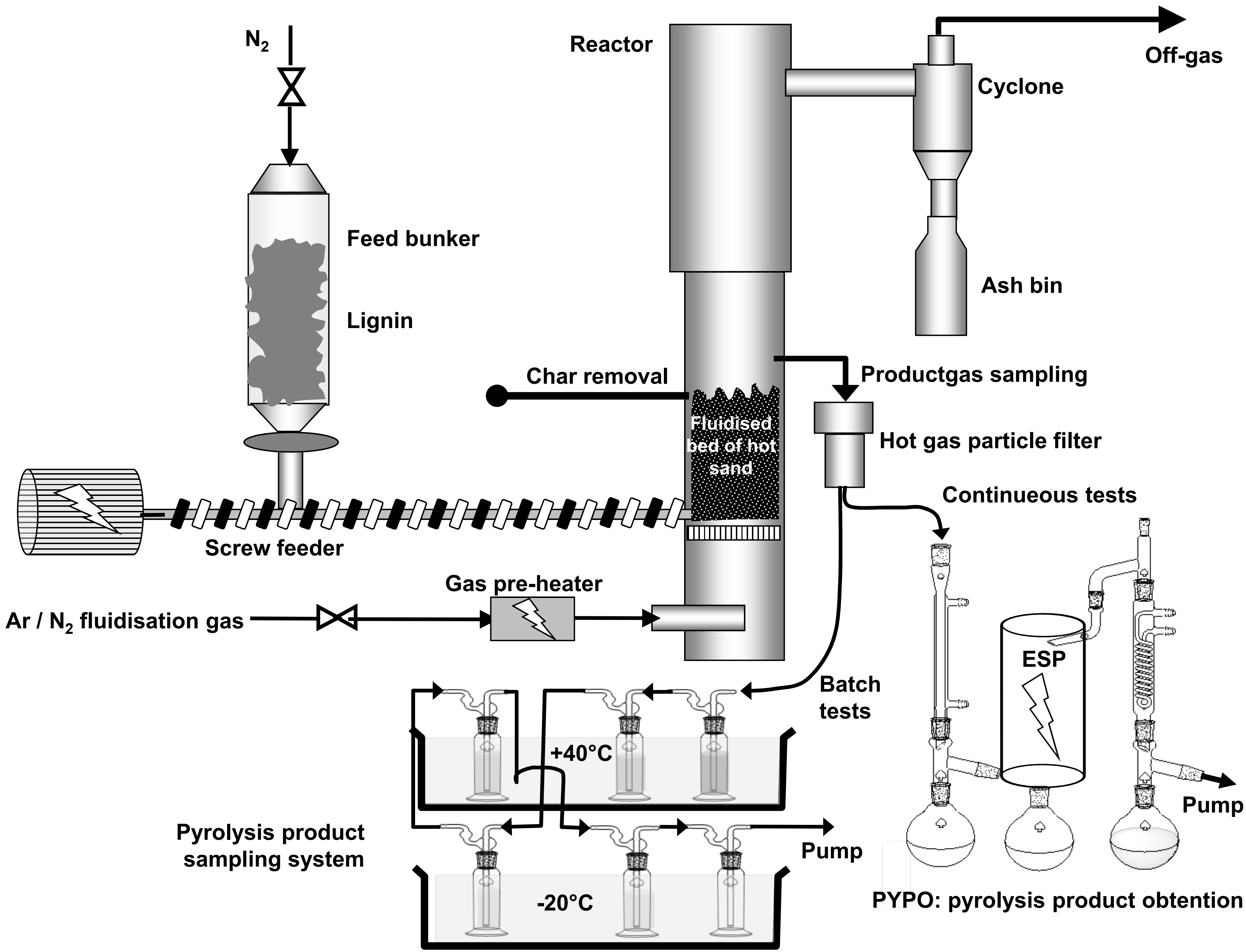


PYPO has been designed in close co-operation with Aston University and captures pyrolysis products (reaction water, aerosols, and organic condensable gases)

The rig features:

- gas/water cooled pre-fractionation **condenser**
- **electrostatic precipitator** at room temperature for the capture of aerosols
- **cold trap** for low-boiling point species.

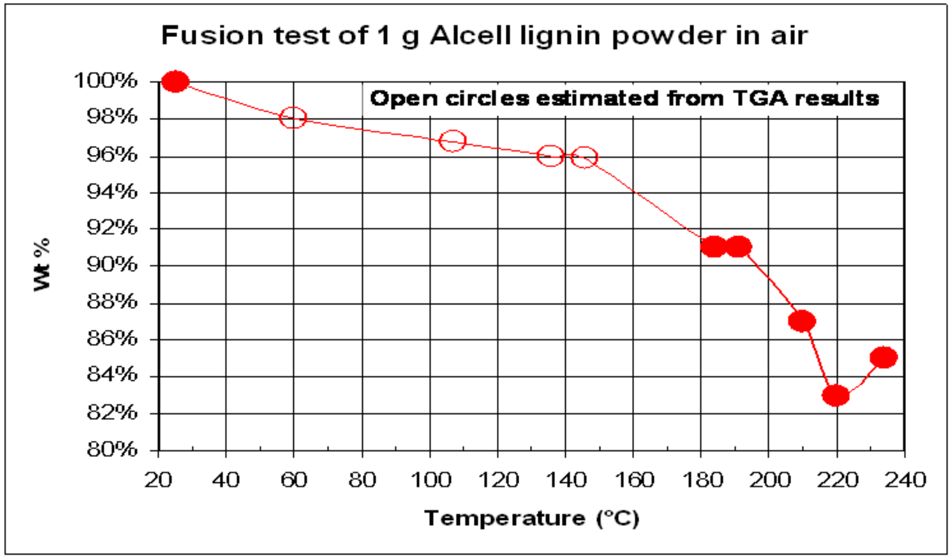
Up to **20 Nl/min** can be sampled from the pyrolysis reactor

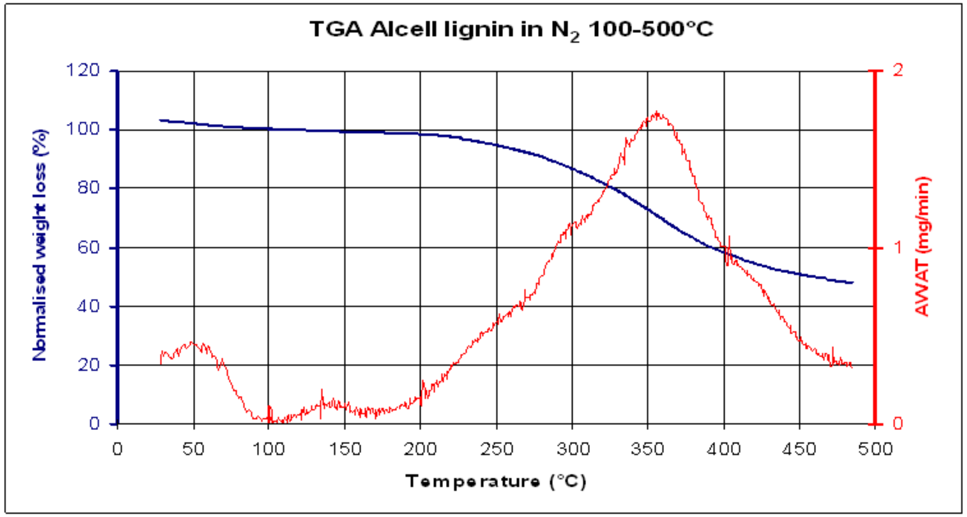


N2
Reactor
Off-gas
Cyclone
Feed bunker
Ash bin
Lignin
Productgas sampling
Char removal
Fluidised bed of hot sand
Hot gas particle filter
Continueous tests
Screw feeder
Gas pre-heater
Ar / N2 fluidisation gas
ESP
Batch tests
+40°C
Pump
Pyrolysis product sampling system
Pump
-20°C
PYPO: pyrolysis product obtention

Results

- **Fusion test**
  Alcell lignin first looses moisture up to 4 wt%, then continues to loose weight until it melts (visual detection) at approx. 180 °C, weight loss ~ 8%. 17 % wt loss at 220 °C
- **TGA**
  Significant wt loss starts at 200 °C. **Max. rate** of degradation at 360 °C. At 500 °C still 45 wt% residual lignin
- **DSC** (phase changes)
  DSC analysis (not shown) shows clear melting tendencies of the lignin powder around 170°C



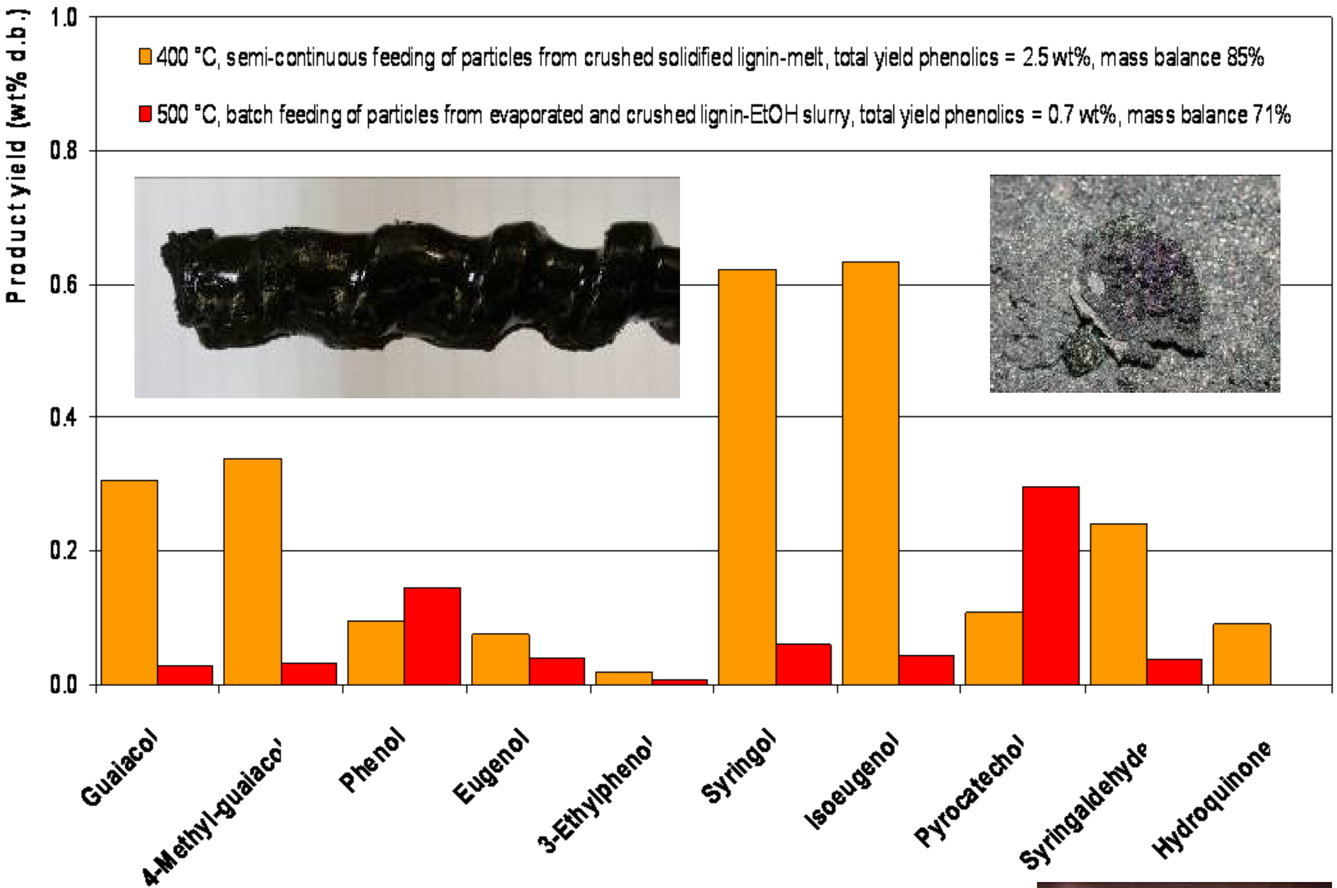


Establish process conditions

Bubbling fluidised bed pyrolysis of Alcell lignin

Initial experiences: bed de-fluidisation due to agglomeration (melting, decomposition)

Alcell lignin pyrolysis degradation products



60% of GC-detected compounds not (yet) identified

At **400 °C** 2.5 % phenolics

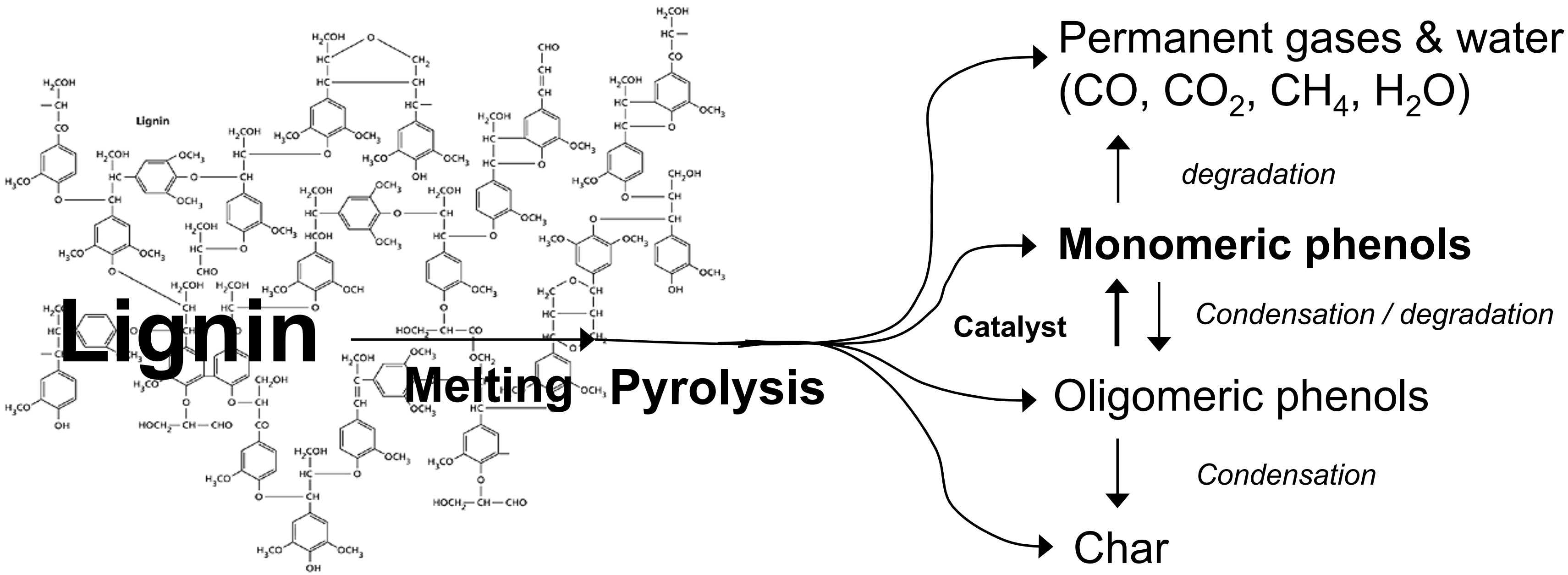
At **500 °C** 0.7 %

Main phenolics at 400 °C: guaiacols, syringols.

At 500 °C more phenol and catechol.

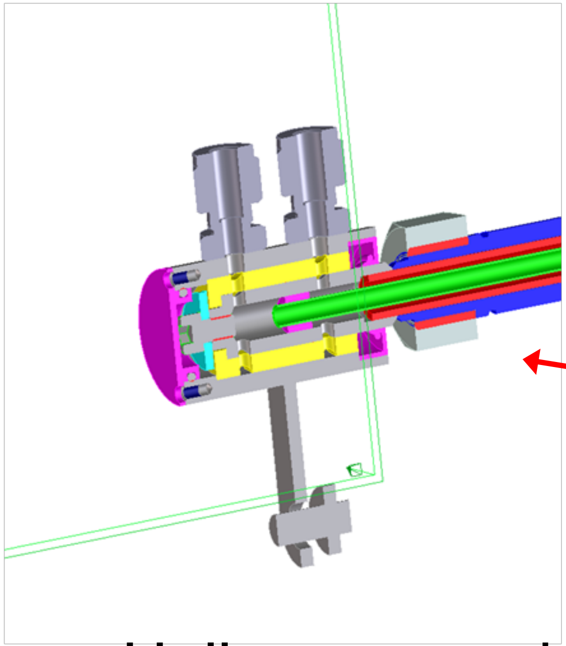
Incomplete conversion due to feeding problems (agglomeration and bed defluidisation)

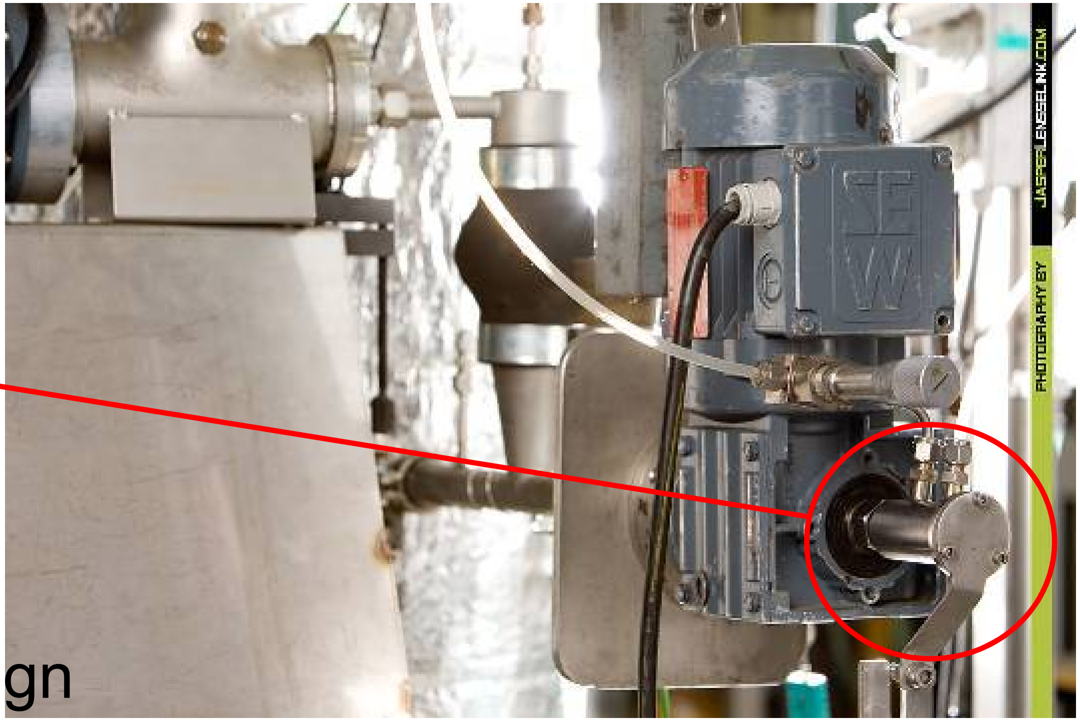
Lignin thermal conversion via pyrolysis: challenges



- First of all a **proper feeding procedure** is required to overcome lignin's thermoplastic behaviour that causes severe operational problems such as screw feeder clogging by molten lignin, agglomeration and subsequent defluidisation of the reactor bed.
- For a maximal conversion of lignin into (monomeric) phenols there is a **narrow window of pyrolysis conditions** such as temperature, heating rate, vapour and solid residence time.
- Use of **catalyst to improve product selectivity and yield**

Improving feeding behaviour: construction of a water-cooled screw feeder



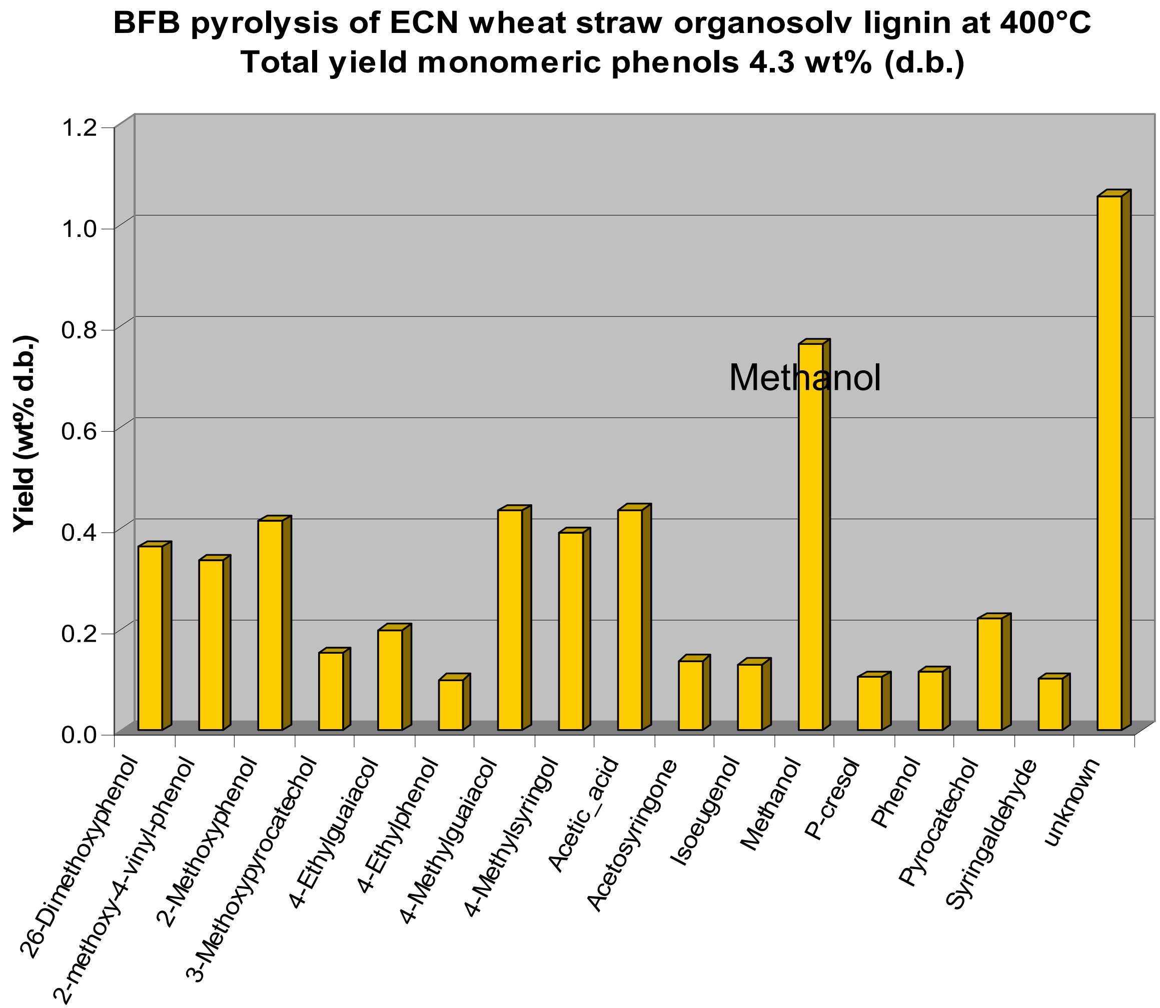


- Hollow screw design

Batch pyrolysis ECN-organosolv lignin

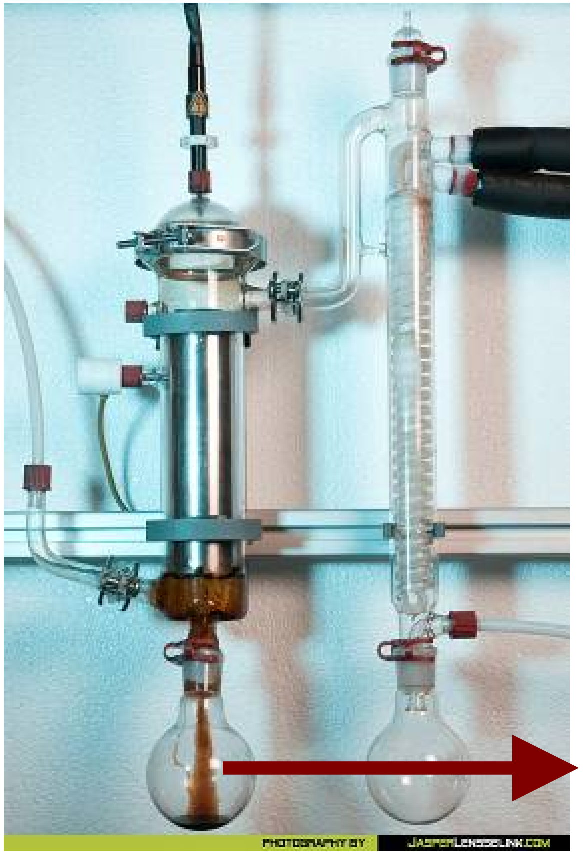
Derived from winter wheat straw,

Small batch (25 g) for BFB pyrolysis trial, pyrolysis at 400°C, mass balance 83%



Continuous catalytic pyrolysis tests

Successful continuous Alcell lignin pyrolysis trials with cooled screw and low feeding rate (150 g/hr) to minimise melting behaviour.



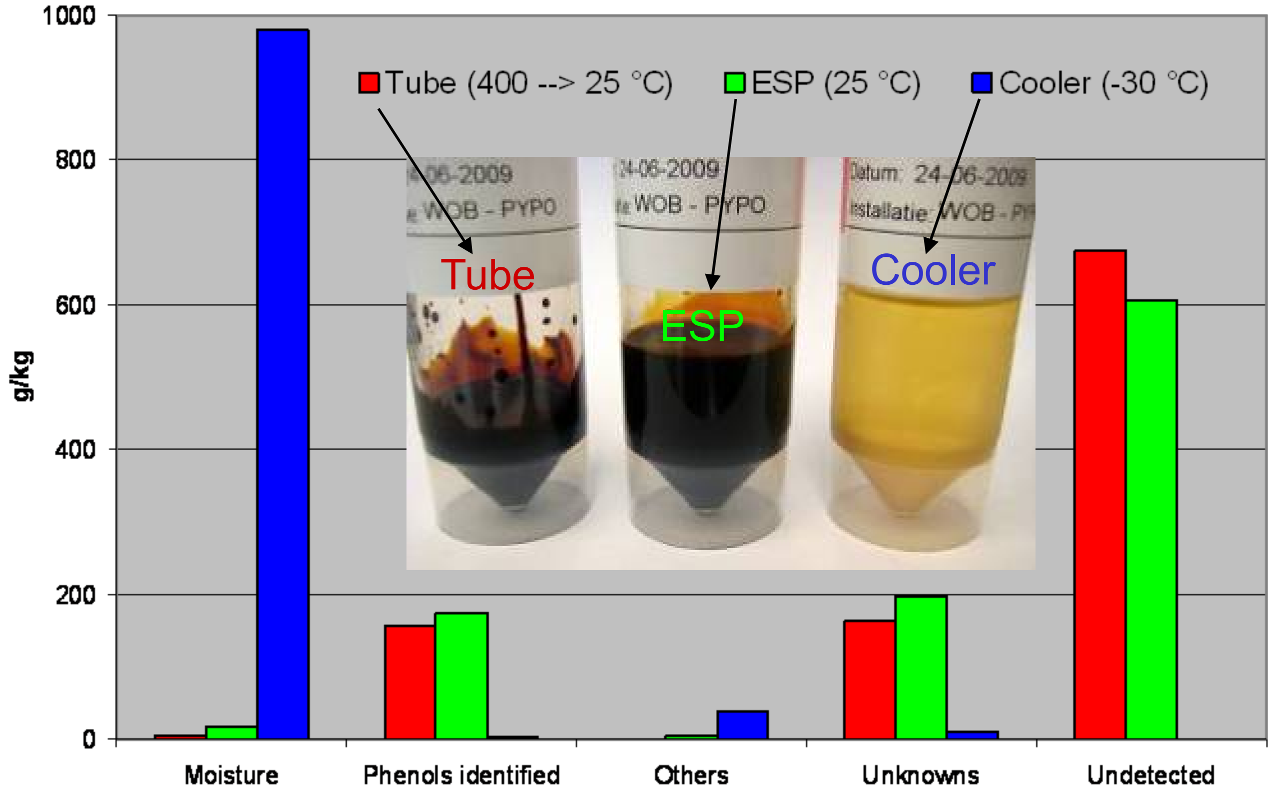
Pyrolytic lignin-oil from Alcell lignin

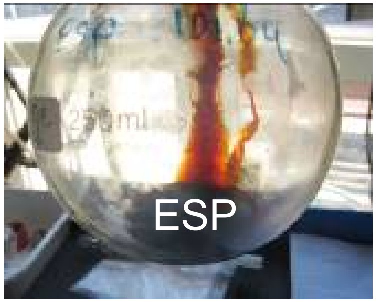
ESP fraction from captured aerosols

Freeze condenser fraction from low-boiling point components

ECN

Results continuous catalytic pyrolysis Alcell lignin (400°C)





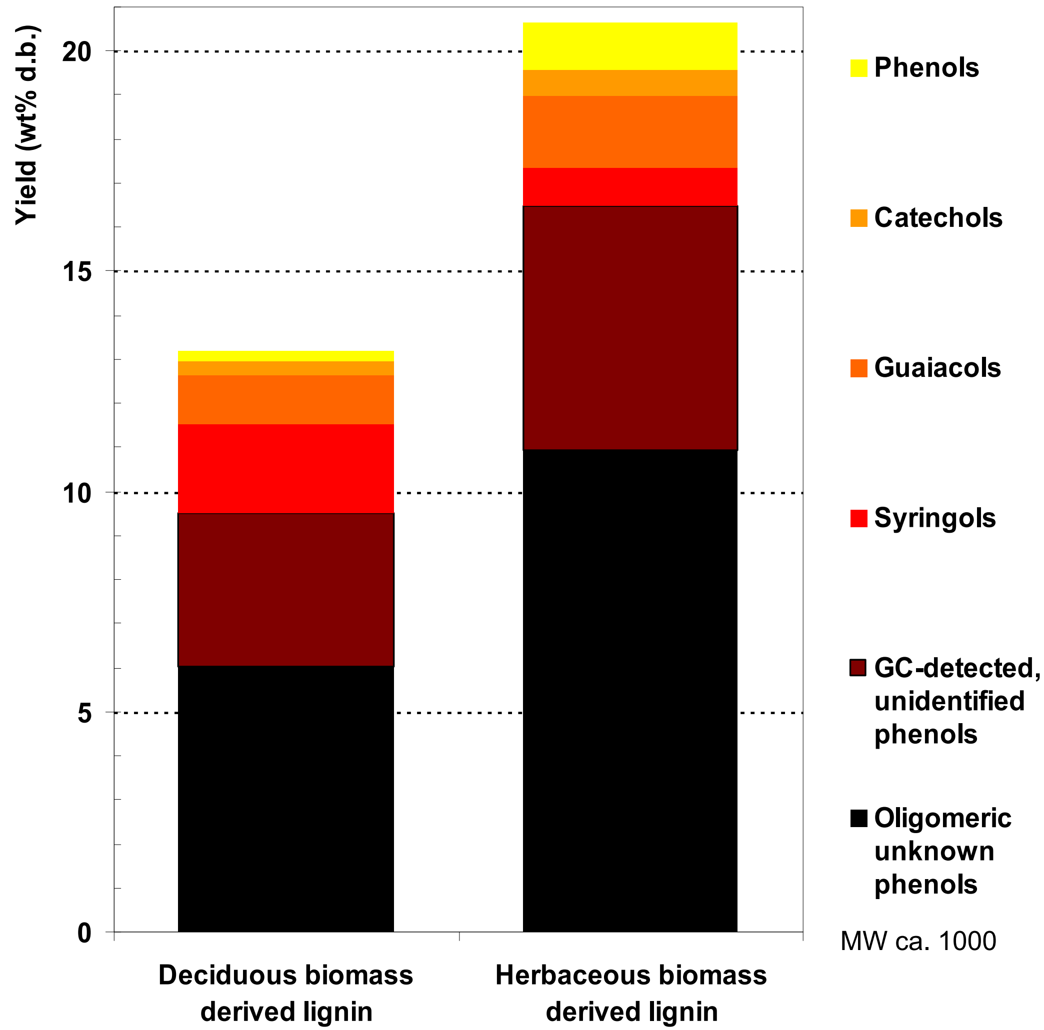
Main product yields (in wt% d.b.):
13 gas, 32 oil (15 phenolics, 3 "light ends", 14 water), 45 char, balance 90 %

Tube = air-cooled condensor.

Comparison Alcell lignin vs. herbaceous derived lignin

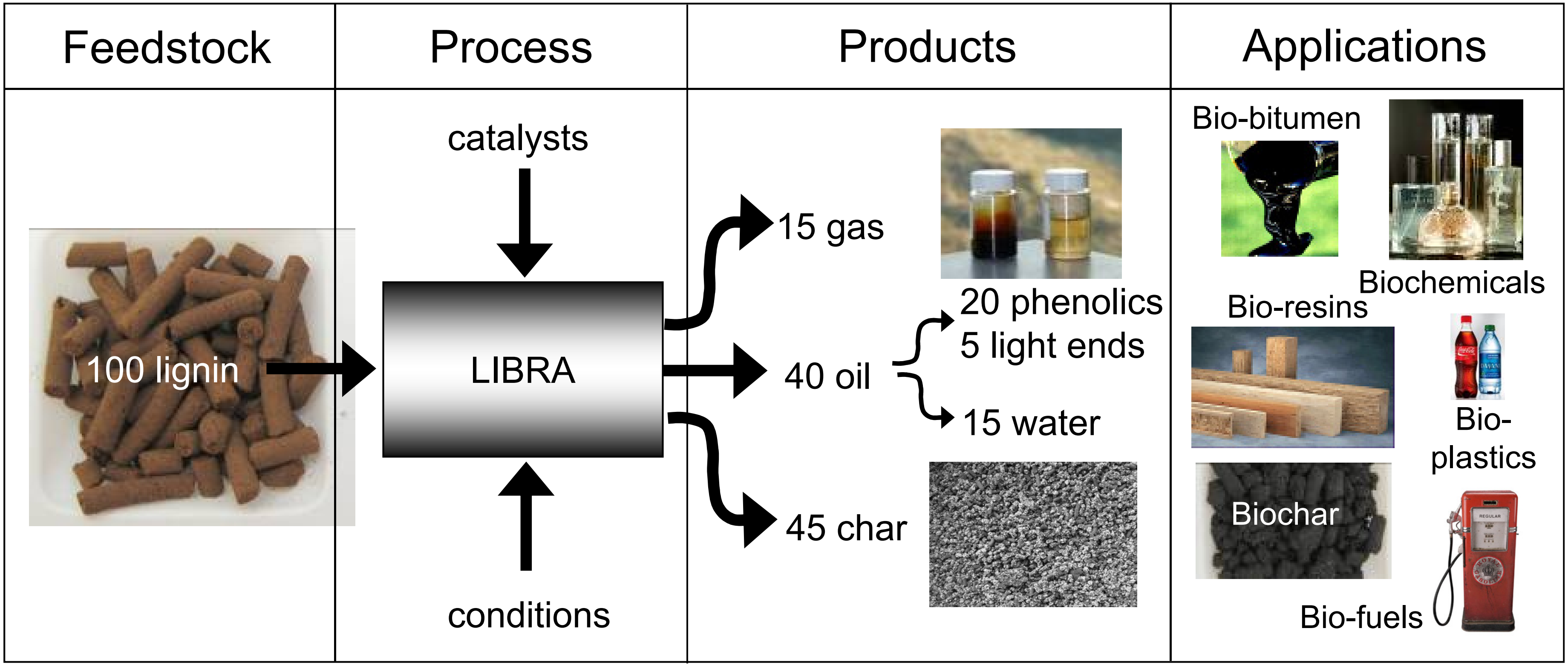
•Bubbling Fluidised Bed catalytic continuous pyrolysis at 400°C yields:

- **13 wt% (d.b.) phenolic** fraction from hard wood Alcell organosolv lignin
- **20 wt% (d.b.) phenolics** from herbaceous lignin from soda pulping of a grass + straw mixture
- Apparently the herbaceous lignin is easier to crack
- Substantial phenolics yields: recent yield improvements up to 40 wt%



MW ca. 1000

Lignin BioRefinery Approach (LIBRA)
Innovative ECN lignin pyrolysis concept



The challenges of feeding, effective thermal degradation and fractionated product collection have (at least partly) been resolved →patent application underway

Conclusions and perspectives

Alcell hardwood-derived lignin can be valorised by bubbling fluidised bed pyrolysis in a phenolic bio-oil and biochar. Typical yields (d.b.) are 15 wt% gas, 40 wt% oil and 45 wt% char. The bio-oil is a mixture of monomeric and oligomeric phenolics, water and low boiling components like methanol.

The **phenolics** can be used to substitute petrochemicals in applications as wood-adhesives/resins, bio-bitumen, chemicals, bio-fuels, etc. The porous **biochar** has potential as soil improver to decrease the amount of fertiliser.

Substantial yield improvement achieved by application of specific catalysts to >20 wt% up to 40 wt% phenolics from lignin

Development lignin biorefinery approach (LIBRA) based on innovations in lignin feeding and catalytic cracking. Patent application is underway.

Thank you for your attention!

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