



BIOSYNERGY

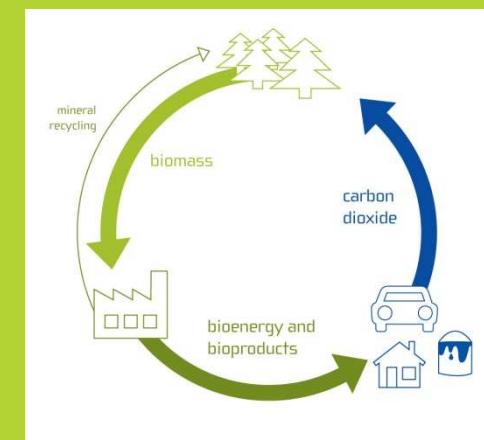


Advanced physical/chemical fractionation

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Workshop of the EU FP6
Integrated Project BIOSYNERGY

November 17th, Reims, France



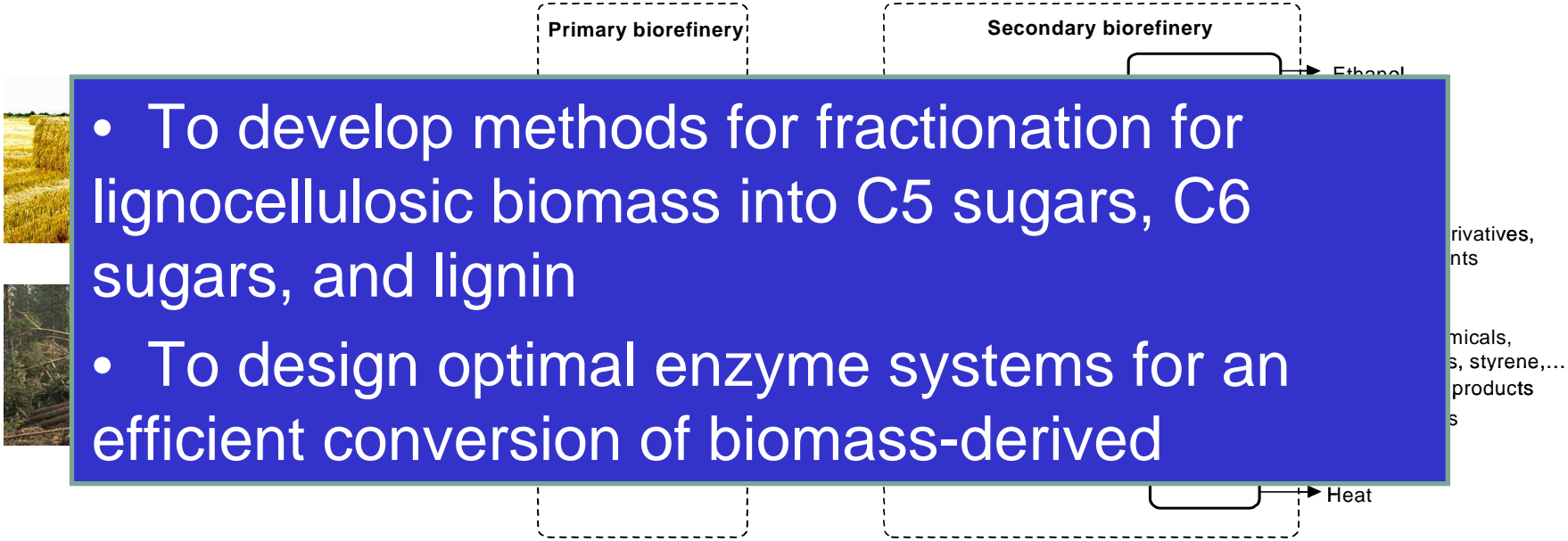


Overview

- Objectives Fractionation work in BioSynergy
- Main processes studied
- Major accomplishments
- Assessment of fractionation routes
- Conclusions



Fractionation of lignocellulosic biomass



Objectives Fractionation:

- Fractionation of lignocellulosic biomass into its **composing fractions** with sufficient quality for production of (bio)chemicals (including lignin).
- **Enhancement (enzymatic) degradability** of cellulose to fermentable sugars.



Main routes studied; partners

- Ethanol/water organosolv



- Organic acid organosolv



- Mechanical/Alkaline fractionation



- Mild fractionation of hemicellulose



- HCl-based hydrolysis



- Enzymatic hydrolysis





Activities

- Proof of concept for fractionation
work on the basis of same lignocellulosic feedstocks
- Exchange of protocols
- Exchange of samples for enzymatic hydrolysis
- Supply of fractionised products to other WP
- Definition and application of technical benchmarks



Ethanol/H₂O Organosolv, ECN



Mech./alk pretreatment A&



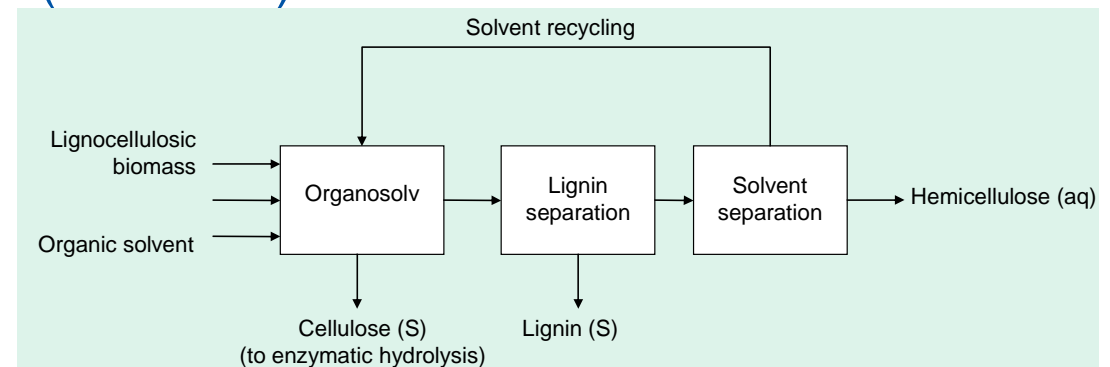
Acid organosolv Pilot plant ARD

Partners: [WUR](#), ABNT, ARD, Bioref, ECN, TUD



Ethanol/water organosolv (ECN)

- Method
 - 160-200 °C, 15-60 min, 5-30 bar.
 - Organic solvent: ethanol (/ acetone)
 - Catalyst: no or H_2SO_4



- Application in Biorefinery
 - Carbohydrates for 2nd generation biofuels or chemicals (furans)
 - High-purity Lignin for conversion into chemicals
 - Remaining solids for energy

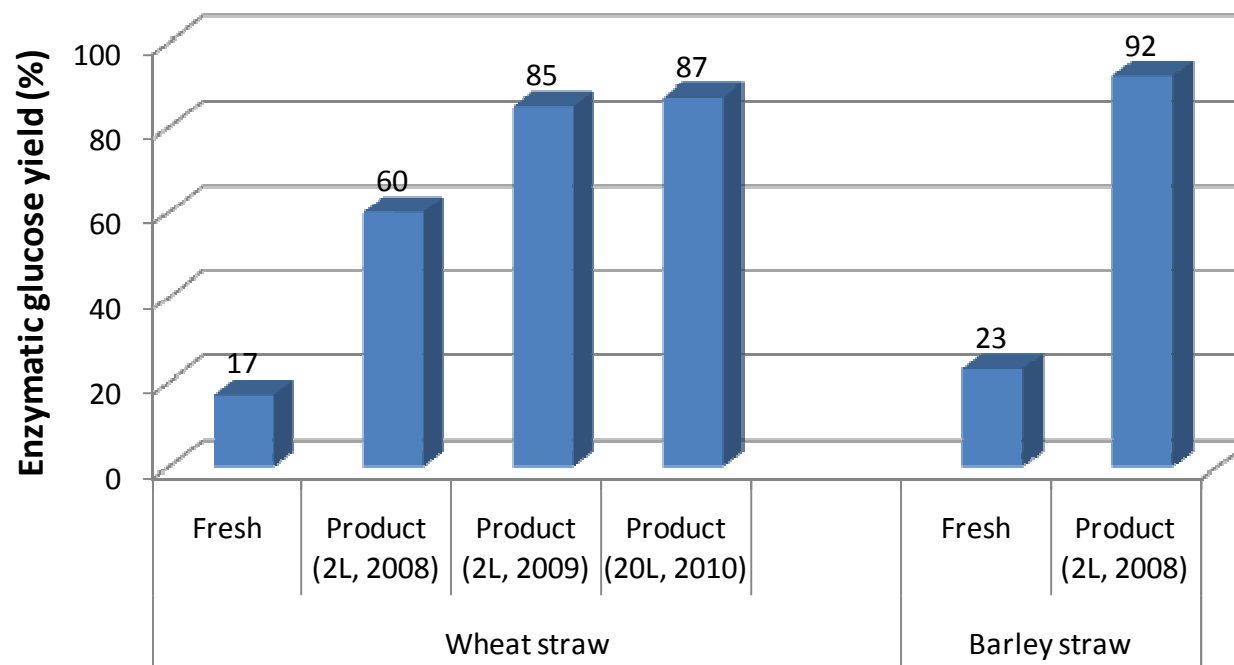


Major Accomplishments EtOH/water organosolv

- Parameters established that govern organosolv fractionation.
- Suitability organosolv fractionation for various feedstocks assessed.
- Scale-up process to 20L batch reactor.
- Application tests of cellulose and lignin.
- Conceptual design completed



Enzymatic digestibility

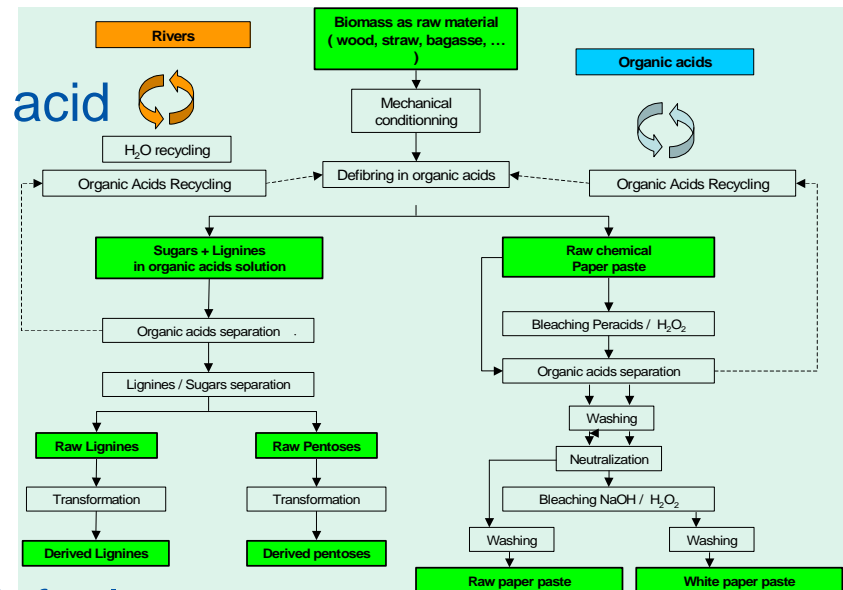




Organic Acid Organosolv (ARD)

- Method

- 105 °C, atmospheric pressure
- Organic solvent: acetic acid, formic acid



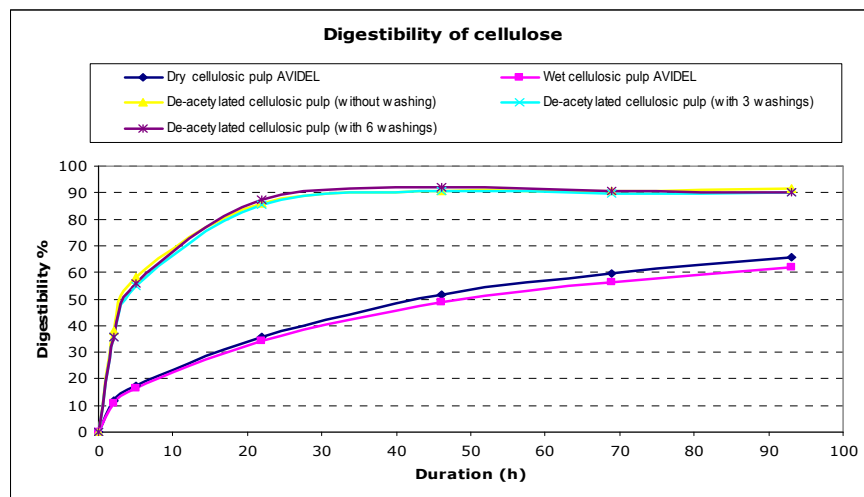
- Application in Biorefinery

- Carbohydrates for 2nd generation biofuels
- High-purity Lignin for conversion into chemicals
- Remaining solids for energy



Major Accomplishments Org. Acid Organosolv

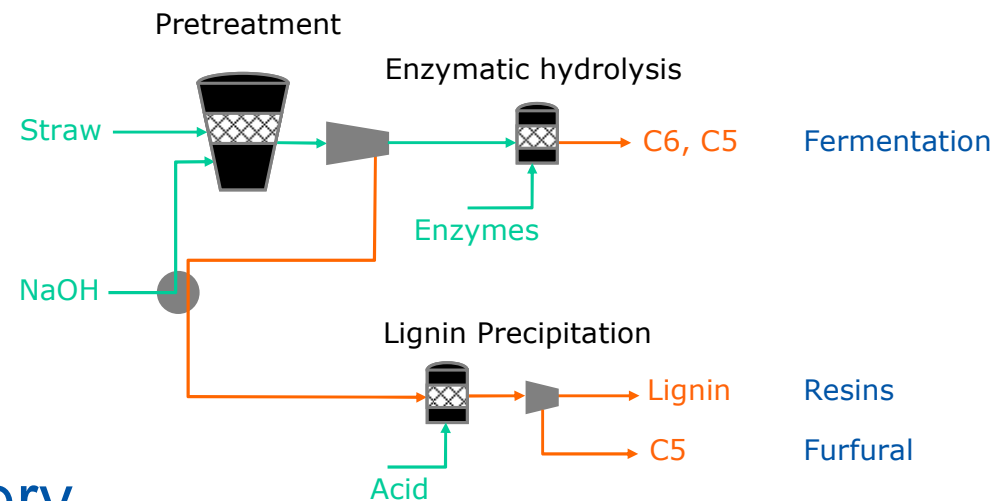
- Improved cellulose digestibility and purity of cellulose
 - By changing acid types
 - By de-acetylation of cellulose pulp
- Experiments performed with woods
- Experiments performed at micro-pilot scale





Mechanical-Alkaline Fractionation (WUR)

- Method
 - 75-100 °C, 1 – 4 h, atmospheric pressure
 - Catalyst: NaOH



- Application in Biorefinery
 - Combined C5/C6 fermentation to alcohols, organic acids
 - Lignin application as phenolic resins
 - Remaining solids for energy



Major Accomplishments Alkaline Fractionation

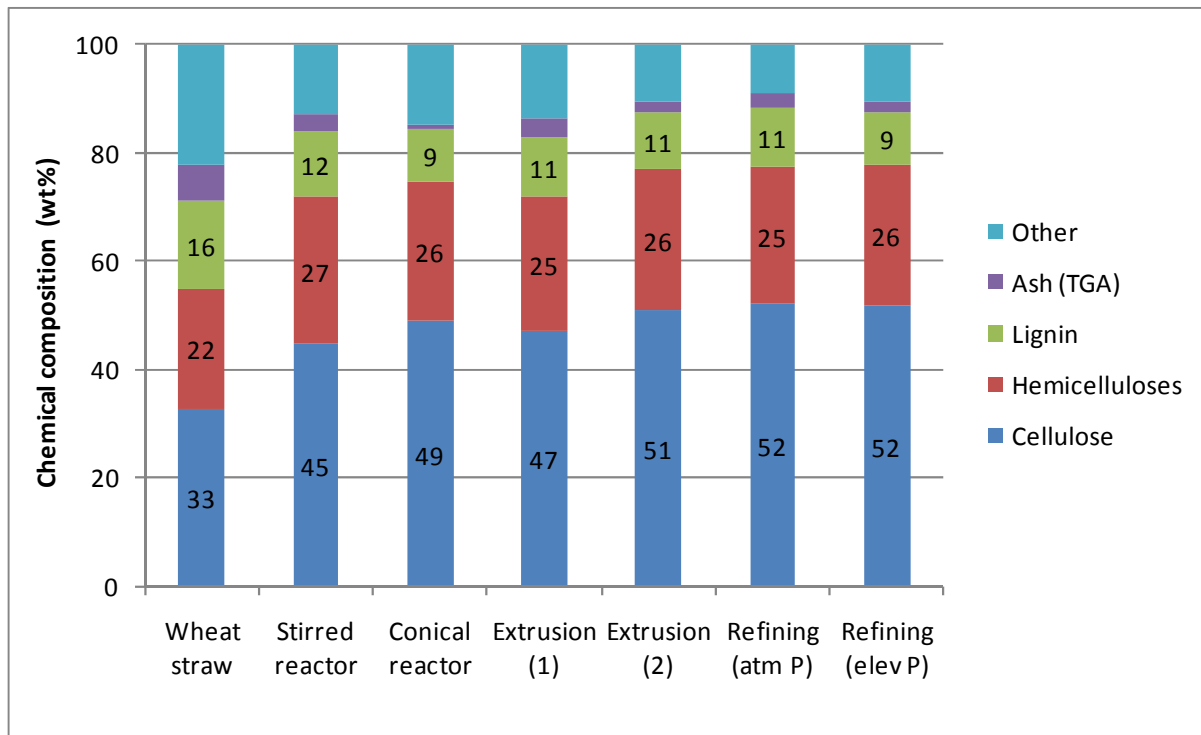
- Effect of processing conditions on chemical composition of pretreated straw and yield
- Enzymatic hydrolysis
 - Effect of dry matter on hydrolysis yields
- Isolation of lignin
 - Effect of processing conditions on black liquor properties
 - Properties of isolated lignins established
 - Application tests of isolated lignin



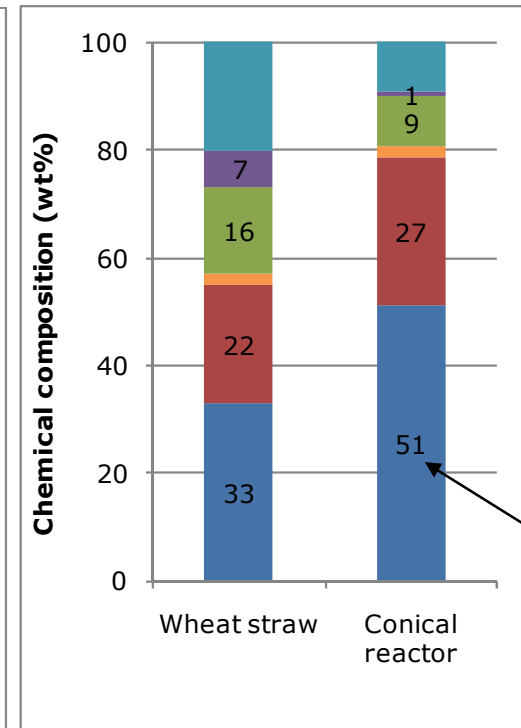
Pretreatment

Efficiency

Lab scale



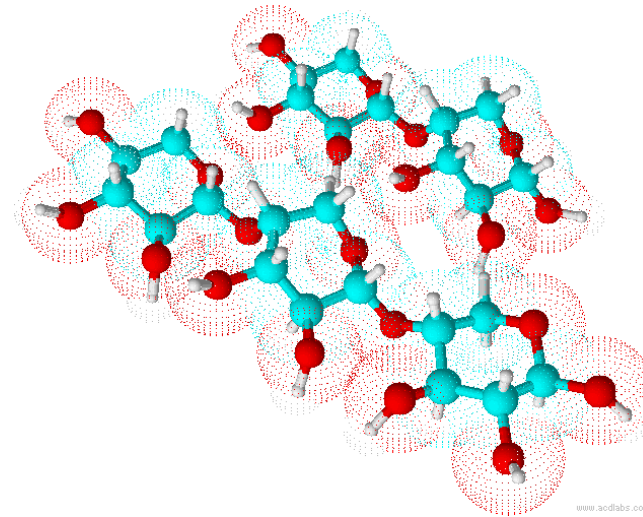
Semi-technical scale





Mild fractionation of hemicellulose (TUDelft)

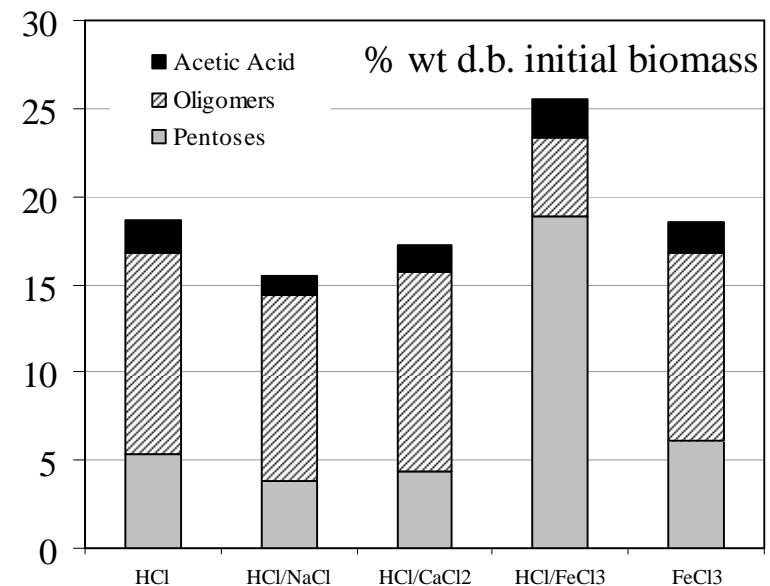
- Method
 - 120 °C, 15-60 min, 5 bar.
 - Catalyst: HCl, FeCl₃
- Application in Biorefinery
 - Selective production of hemicellulose-derived carbohydrates
 - Further conversion into furfural, xylitol, surfactants etc
 - Solid residue for enzymatic conversion, or further separation routes
 - Remaining solids for energy





Major Accomplishments hemic. fractionation

- Proof of principle mild ($T < 120\text{ }^{\circ}\text{C}$) fractionation of hemicellulose
 - Relatively simple process, low-cost operation expected
 - Cellulose remains intact after C5 extraction and is free of minerals
 - Enzymatic degradability of cellulose needs improvement





HCl-based Acid hydrolysis (BioRef)

- Method
 - 32% - 37% HCl, room temperature

- Application in Biorefinery
 - Combined C5-C6 fermentation to alcohols
 - Applications of lignin products in hardboards
 - Remaining solids for electricity and heat



Major Accomplishments Acid hydrolysis

- Improved direct saccharification with a two-step strong hydrochloric acid application
 - No enzymes required
- Remaining lignin with good thermal properties
- Fractionation method is applicable for all feedstocks



Enzymatic hydrolysis (ABNT)

Objectives

- Achieve High productivity strains
- Increased enzyme effective activity which makes a reduction possible of enzyme dosage below 5mg/ g cellulose during enzymatic hydrolysis
- Production of enzymes at competitive cost



Major Accomplishments Enz. hydrolysis

- New supplemental enzymes developed
- Trials performed at 20% wt/wt
- Increased enzymatic activity of commercial enzyme mixes when supplemented
- Yield of SSF with new enzymes 44 g/L ethanol



Benchmarks for evaluating Fractionation

- Delignification
- Lignin purity, quality
- Hemicellulose hydrolysis
- Enzymatic degradability of cellulose
- Fermentability of glucose obtained from cellulose

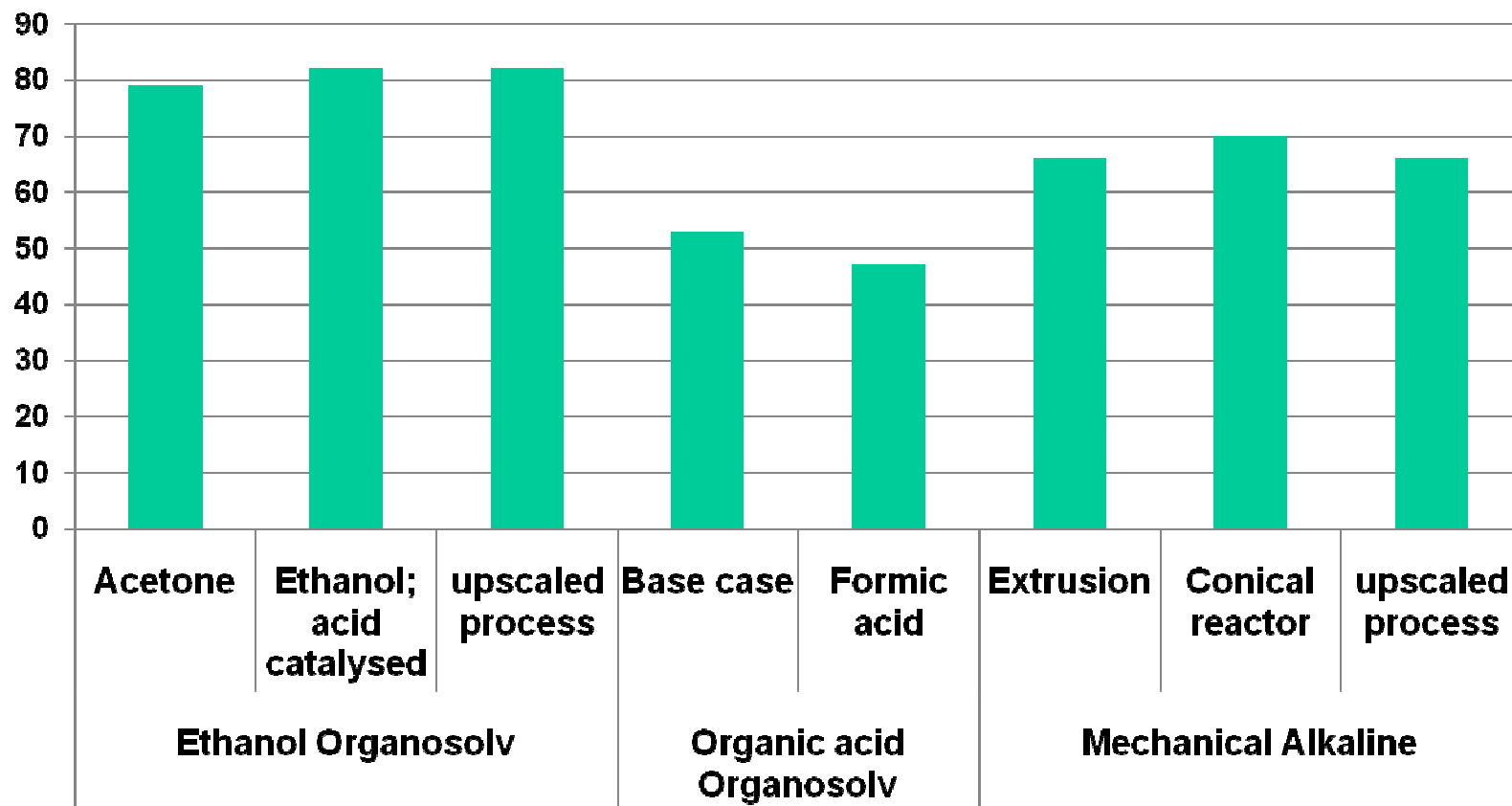


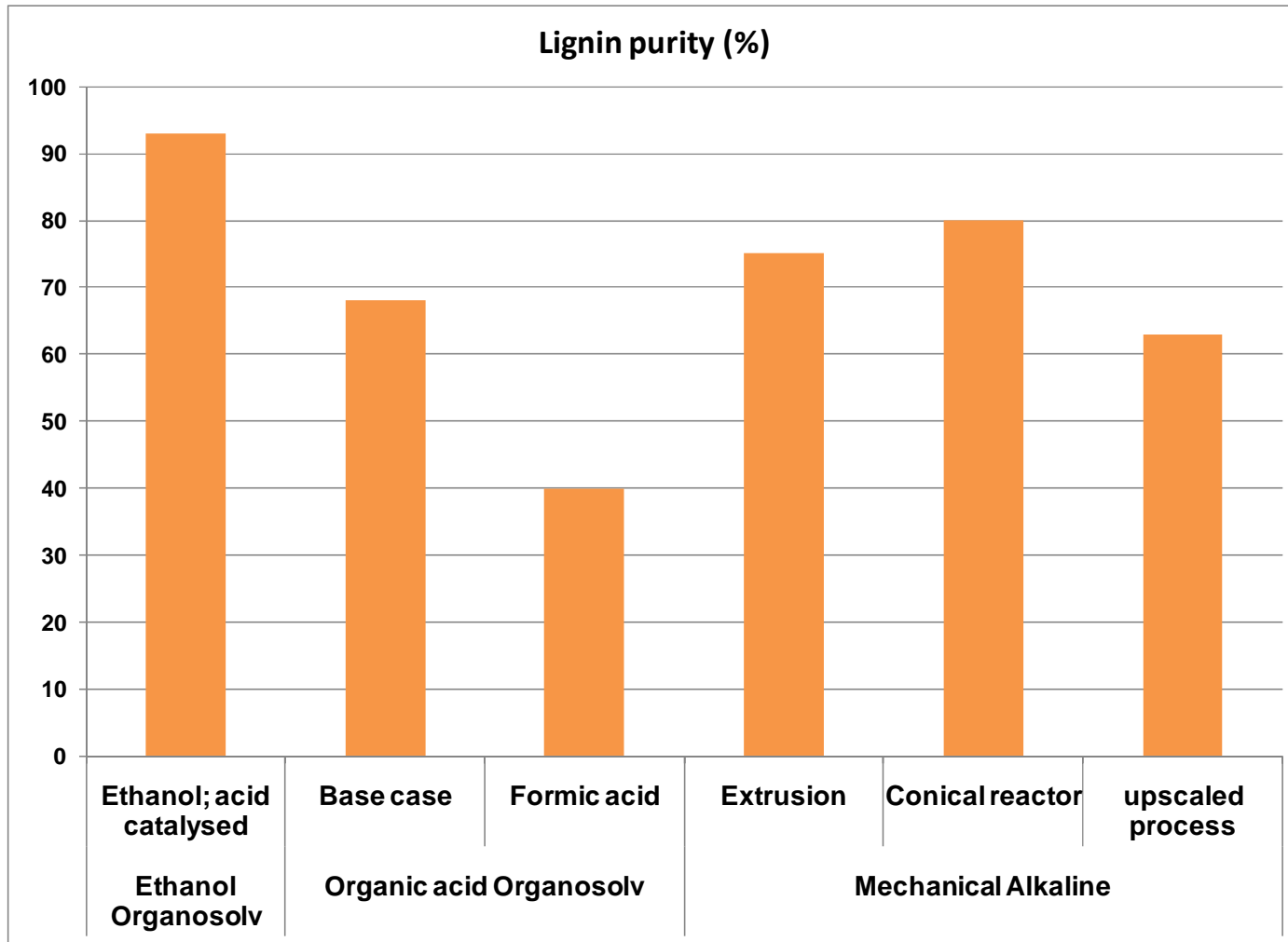
Results benchmarking (wheat straw)

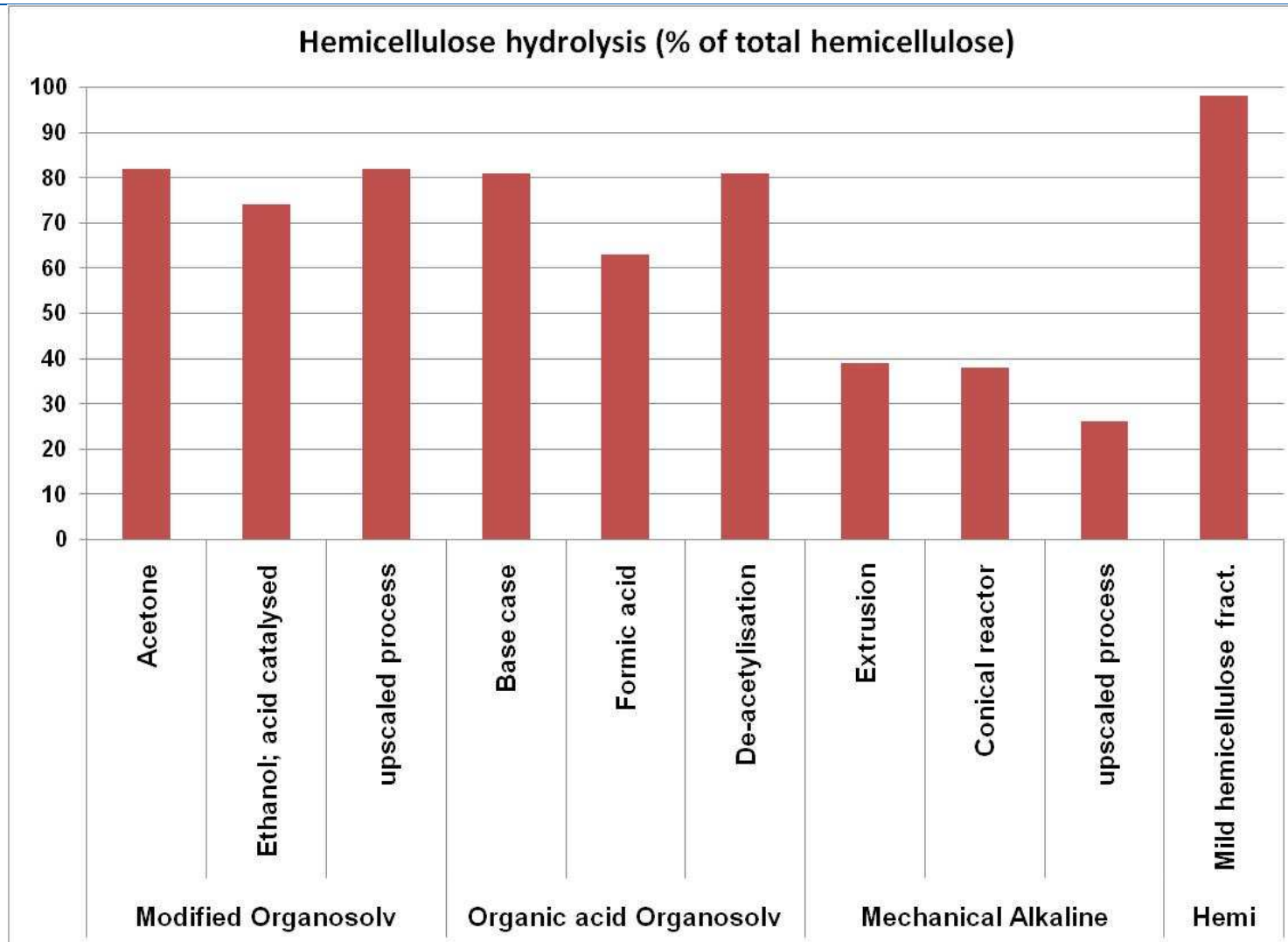
		Delignification	Lignin purity	Hemicellulose hydrolysis	Cellulose degradability
EtOH Organosolv	Acetone	79		82	87
	Ethanol; acid catalysed	82	93	74	85
	upscaled process	82		82	87
Organic acid Organosolv	Base case	53	68	81	52
	Formic acid	47	40	63	76
	De-acetylisation			81	90
Mechanical Alkaline	Extrusion	66	75	39	100
	Conical reactor	70	80	38	89
	upscaled process	66	63	26	62
Hemicell fractionation	Mild hemicellulose fract.			98	40



Delignification (% of total lignin)









Main conclusion

- **All technologies lead to significant fractionation into composing elements C5, C6 sugars and lignin**
 - High cellulose degradability
 - Differences in hemicellulose hydrolysis, and lignin yield and – characteristics
 - Often trade-off between desired effects
- **Fractionation technologies need to be optimized toward a particular goal**
 - An integrated approach feedstock-process-endproduct is therefore required!



Remaining Issues

- Feedstock flexibility
- Development of large scale lignin applications
- Recycling of reactants
- Improving enzymes, esp. for upscaling
- Use of organic acids in stead of mineral acids
- Developing continuous reactor systems for fractionation



Acknowledgments

- ECN: Wouter Huijgen, Hans Reith
- A&F: Paulien Harmsen, Bram Sperber, Steef Lips
- ABNT: Laura Bermudez, Reyes Capote
- ARD: Frederic Martel, JC Rambourg, A. Bresin
- Biorefinery.de: Petra Schoenicke
- TuDelft: Gianluca Marcotullio, Wiebren de Jong

This work was performed in the context of:

