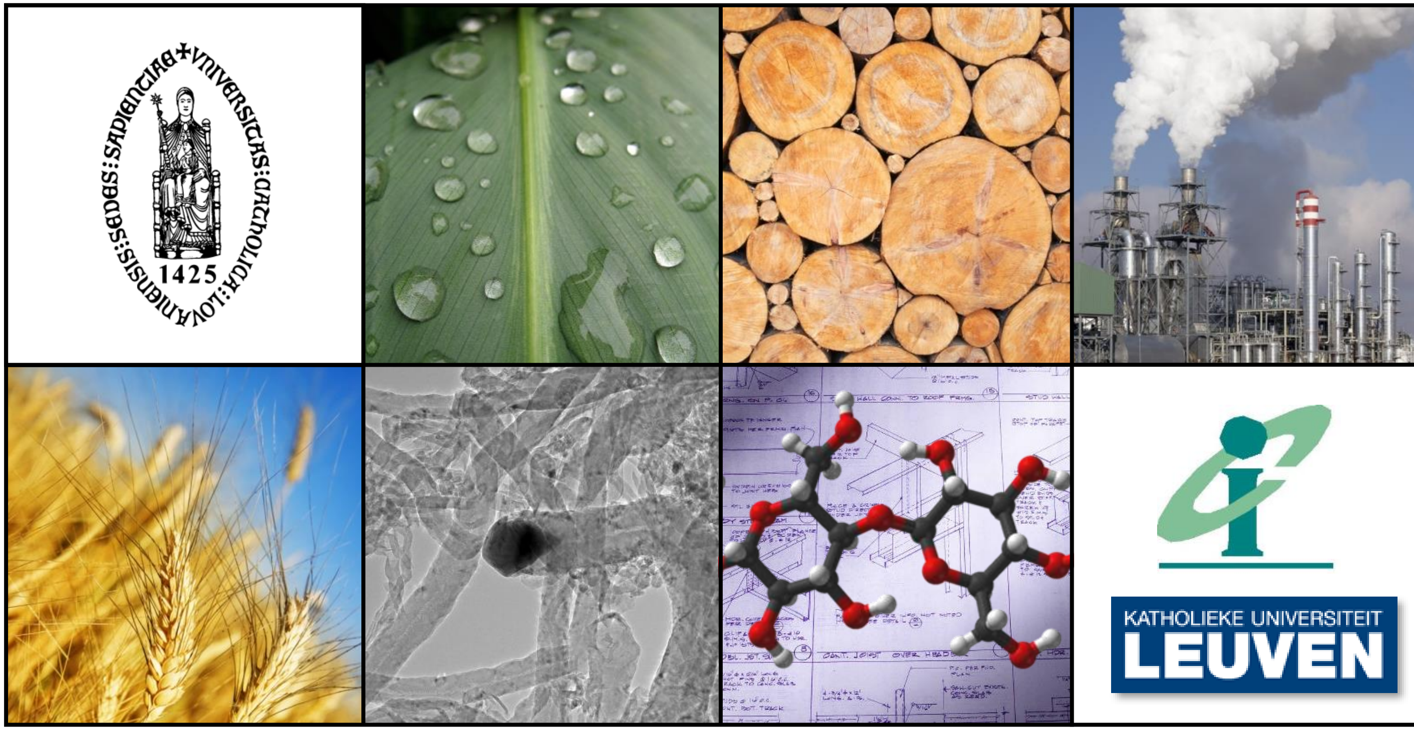

KATHOLIEKE UNIVERSITEIT
LEUVEN
lignoCOST
1425
LIGNOCOST, Wageningen



APPLYING PRODUCTION AND USAGE OF NEW LIGNIN STREAMS

CENTER FOR SUSTAINABLE CATALYSIS AND ENGINEERING
UNIVERSITY OF LEUVEN
BELGIUM

bert.sels@kuleuven.be
http://www.sels-group.eu/

OUTLINE

Lignocellulose and its biorefinery

Lignin challenge ?

- **From carbohydrate to lignin-centered biorefinery**

Reductive Catalytic Fractionation - RCF

- **What is RCF biorefinery ?**
- **Role of catalysis ?**
- **Upscale challenges & initiatives at KULeuven (BIOCON)**

Refined fractions and downstream products

Conclusion & Perspectives

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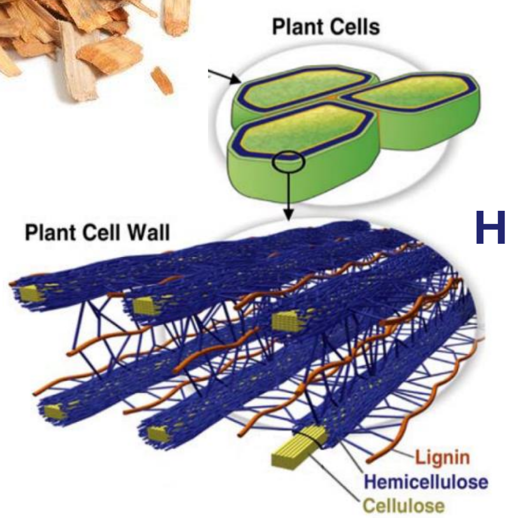
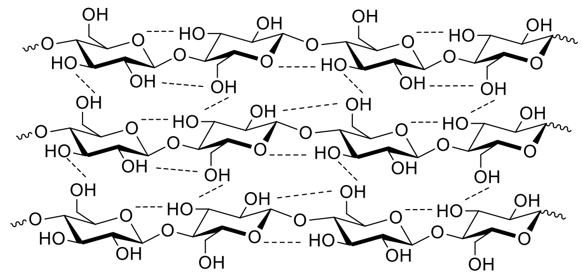
Biorefining lignocellulose

Lignin Chemistry Ed. Serrano, Luque, Sels *Topics in Current Chemistry Collection*, Springer 2020.

LIGNOCELLULOSE AND ITS BIOREFINERY

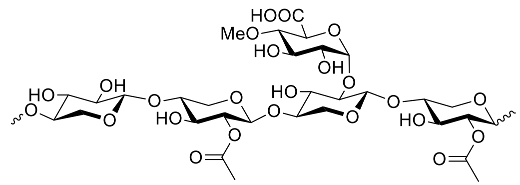


Cellulose



- 40-60 wt%
- Semi-crystalline
- Rigid fibers

Hemicellulose



- 10-35 wt%
- Different C5-C6 sugars
- *'Family'* of polymers
- Amorphous

…and Lignin

- *'Glue'*
- 15-30 wt%

Lignin Chemistry Ed. Serrano, Luque, Sels Topics in Current Chemistry Collection, Springer 2020

LIGNOCELLULOSIC BIOREFINERY

Unraveling lignocelullosics into "useful" streams

Syngas

Gasification in oxygen – upgrade by (existing) C1 chemistry (CH_3OH, Methanol to hydrocarbons, Fischer-Tropsch, …)

Bio-oil

(hydro-)Pyrolysis in inert or (supercritical) solvent (optionally with catalysis) – novel refinery of oxygenates from both sugar and lignin part – further upgrade to aromatics, olefins, hydrocarbons (biofuel), …

Carbohydrate pulps

Solvolytic or hydrolytic fractionation *(water, organic polar solvent)* – sugar chemistry, fermentation, … .

?? Lignin: kraft, alkali, hydrolysis, organosolv

High T
Original structure loss
Full solid biomass conversion

↓

Low T
Original structure retainement
Separating products from carbohyrdate and lignin

LIGNOCELLULOSE AND ITS BIOREFINERY

Cellulose
Hemicellulose
Lignin ?

Polymers
Lubricants
Coatings
Adhesives
Paint
Fuels
...

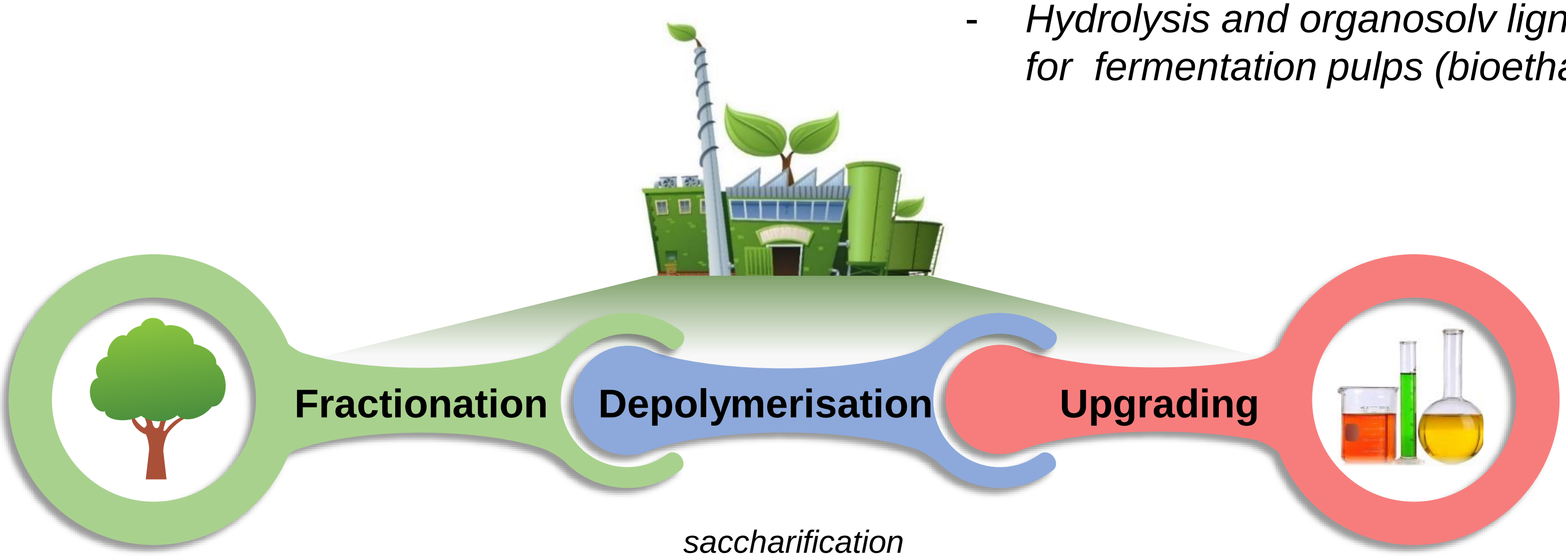
Bioaromatics

Lignin Chemistry Ed. Serrano, Luque, Sels Topics in Current Chemistry Collection, Springer 2020

From carbohydrate centered to *lignin-first* biorefineries

THE CLASSIC SUGAR-CENTERED BIOREFINERY

Examples:
- *Production of paper (e.g., Kraft)*
- *Hydrolysis and organosolv lignin for fermentation pulps (bioethanol)*



saccharification

fermentation

Biomass → Cellulose → Sugars → Bioethanol

Biomass → Hemicellulose → Sugars → Bioethanol

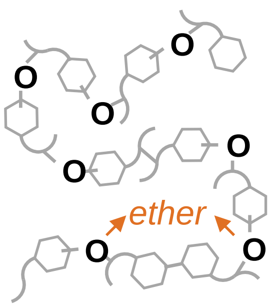
Biomass → Lignin 🔥

Acid, Base
Enzymes
T
Contact time
Solvent

Schutyser *et al*. ChemSocRev 2018
Renders *et al*. EES 2017

LIGNIN IN A CLASSIC BIOREFINERY ?

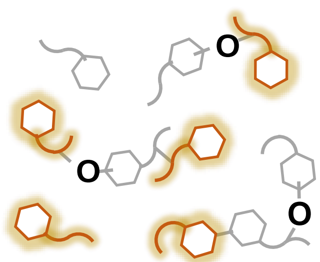
Biomass → Cellulose / Hemicellulose / **Degraded** lignin



Native or in planta lignin (e.g., b-O-4)

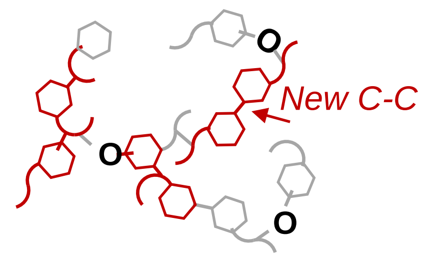
Solvolytic Cleavage of part of the ethers

Disentaglement from sugars



Reactive (olefinic or carbonyl containing) lignin fragments

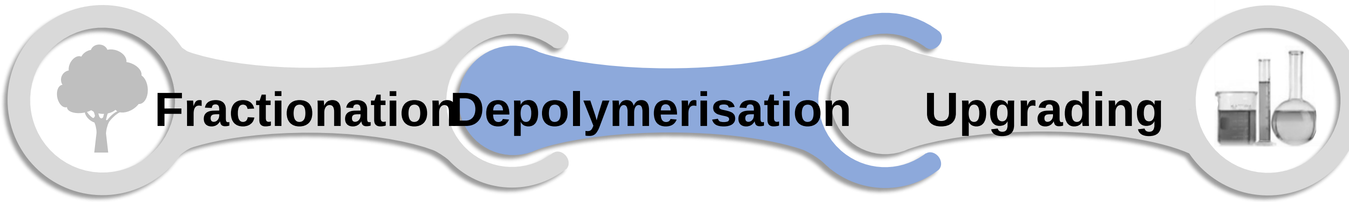
Condensation with new C-C bond formation



Isolated lignin precipitates

Schutyser *et al*. ChemSocRev 2018
Renders *et al*. EES 2017

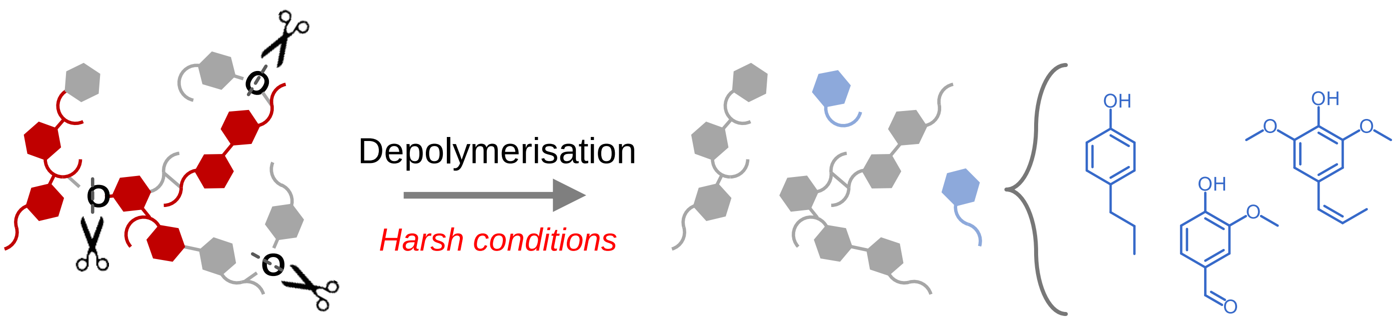
THE LIGNIN CHALLENGE



Degraded lignin → **Phenolics**

(low functionalisation)

(low yield for low MW and monomers)



Isolated lignin *Depolymerised lignin* *Examples*

Refunctionalize the available commercial lignin

Schutyser *et al*. ChemSocRev 2018
Renders *et al*. EES 2017

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LIGNIN-FIRST STRATEGIES

Lignin protection strategies while dissolving the pulp in strong acid (step 1) + selective depolymerisation of the isolated lignin (step 2).

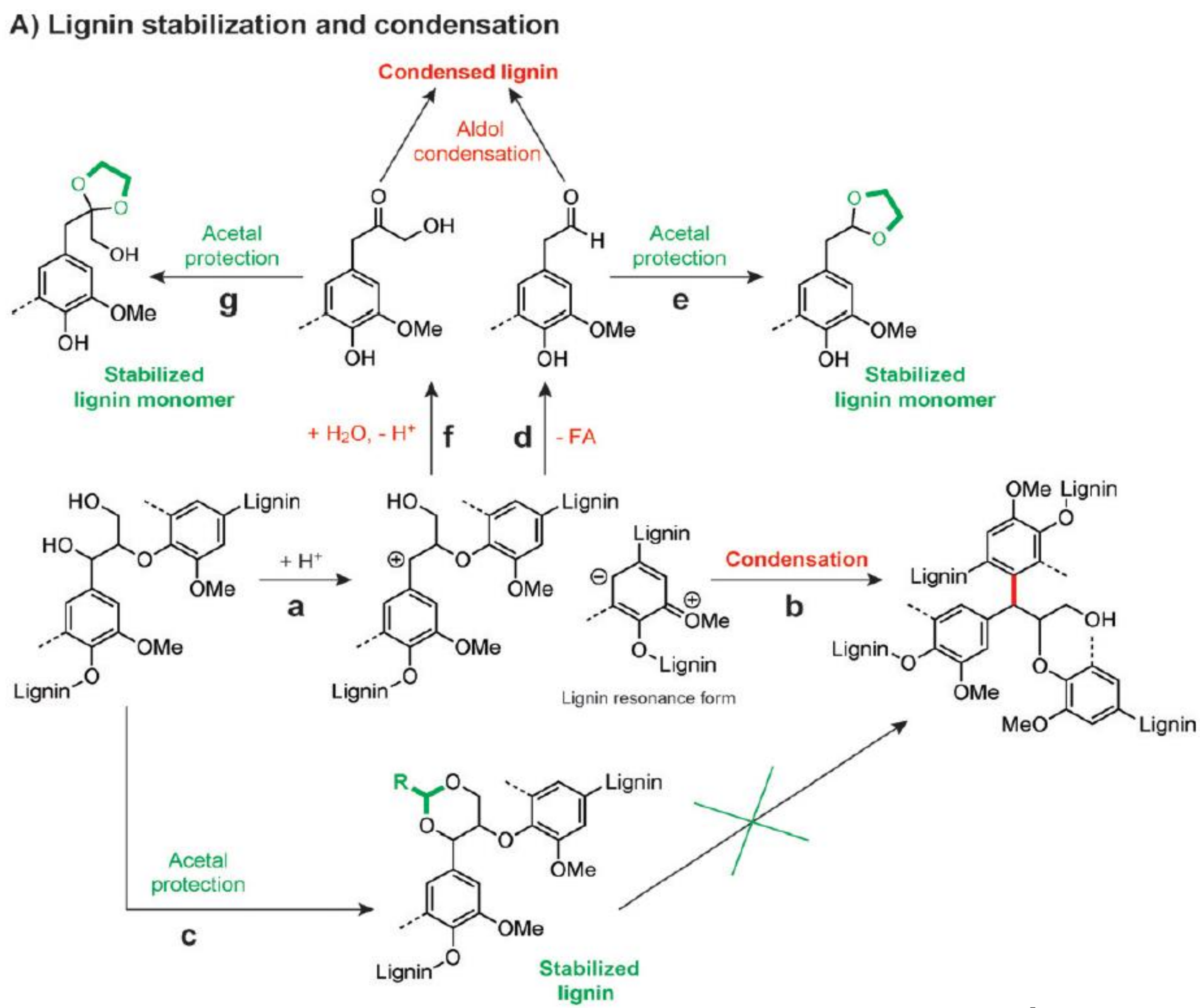
- Aldehydes (Lutherbacher *et al*.)

Chemical stabilization of the reactive intermediates

- Aromatics alkylation (phenolation) – *high MW, high phenol content*
- Glycols (Barta *et al*.)
- Reductive chemistry – monomers to *low MW* (e.g., Song *et al*., Abu-Omar *et al*., Rinaldi *et al*., Barta *et al*., Sels *et al*.)

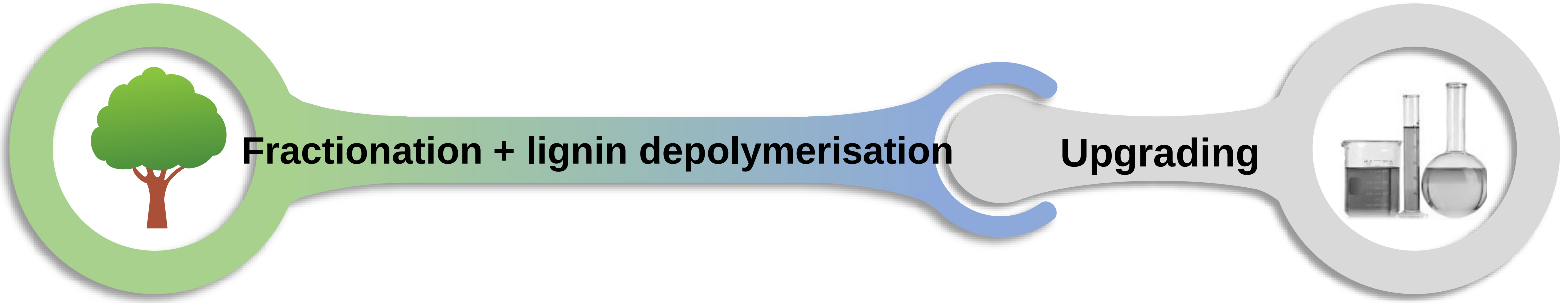
EES 2020 (guidelines for lignin-first)
Angewandte Chemie 2020

EXAMPLES OF LIGNIN STABILISATION IN CHEMISTRY LANGUAGE



Angewandte Chemie 2020

A NEW CONCEPT:
LIGNIN-FIRST BIOREFINERIES

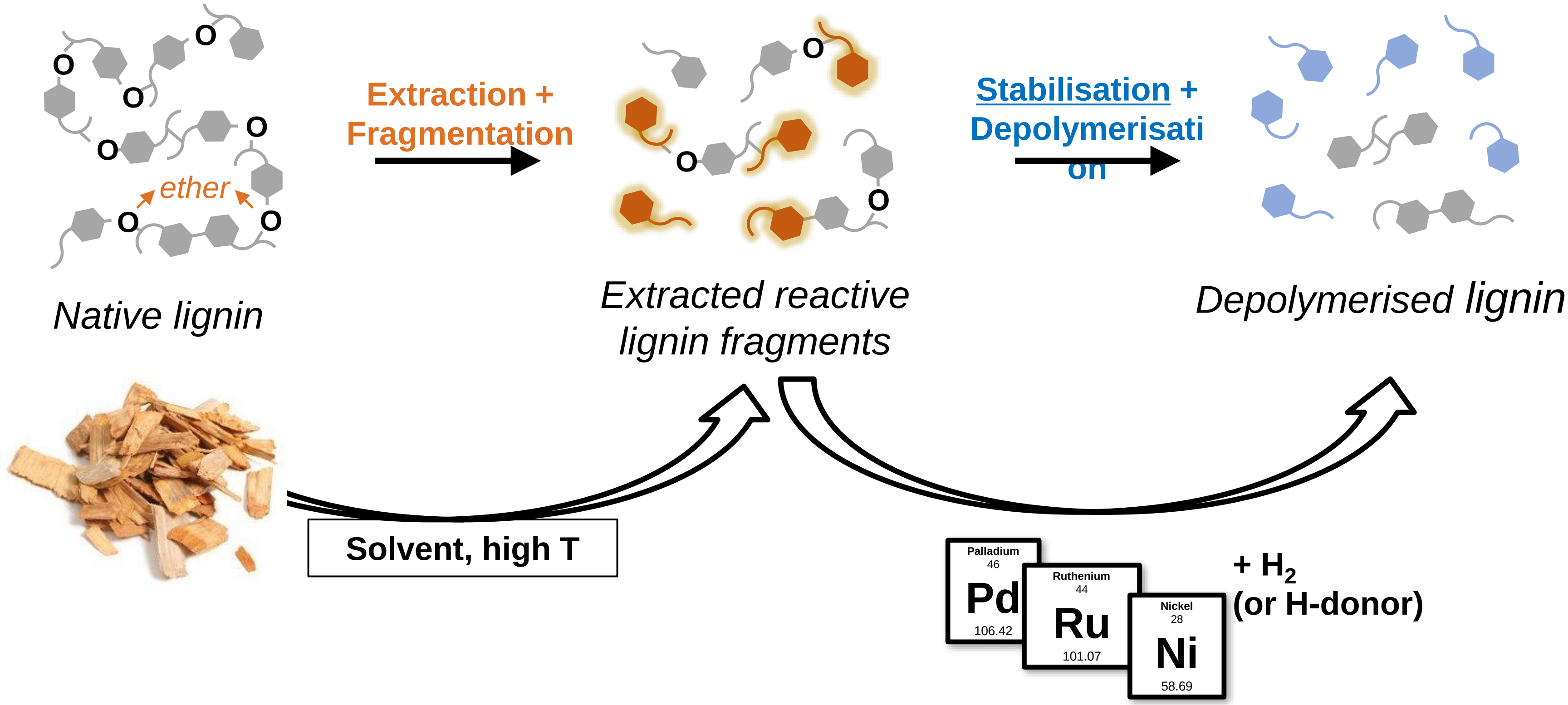


Lignin–first *Biorefineries*

'innovative lignin streams'w.r.t novel unique structures and properties

REDUCTIVE CATALYTIC STABILISATION: CONCEPT

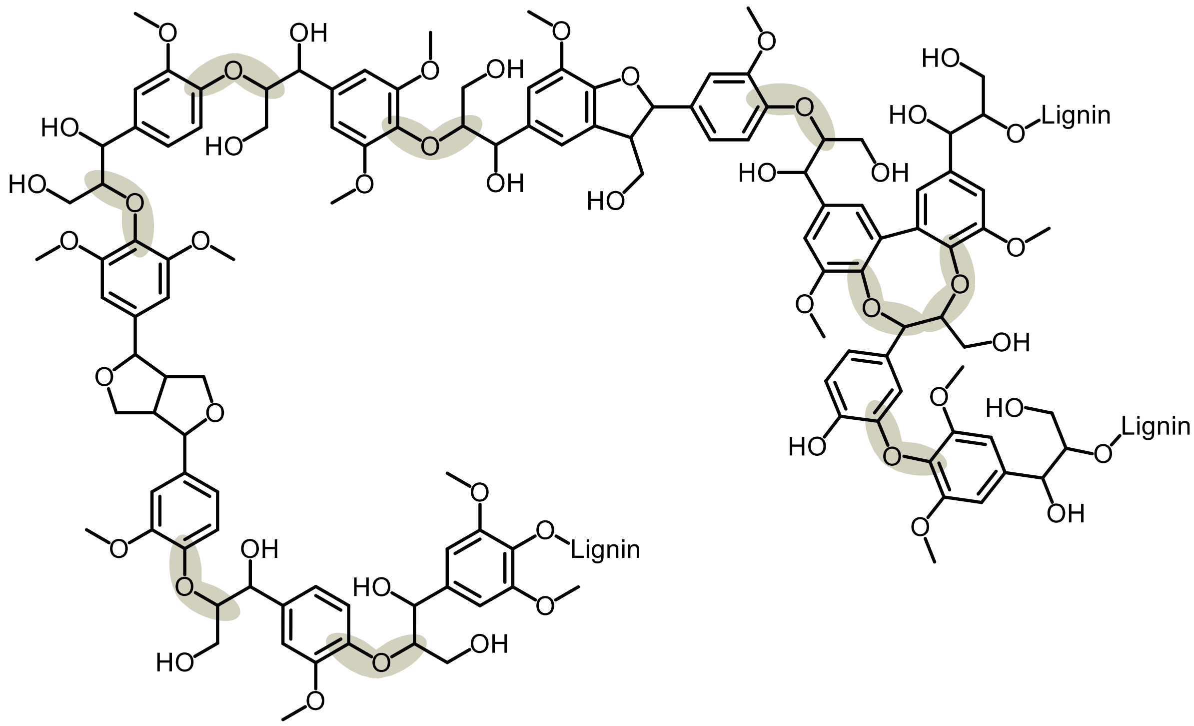
Reductive Catalytic Fractionation (RCF)



catalyst

Renders *et al. Energy Environ. Sci.*, **2017**
Lignin first guidelines EES 2020

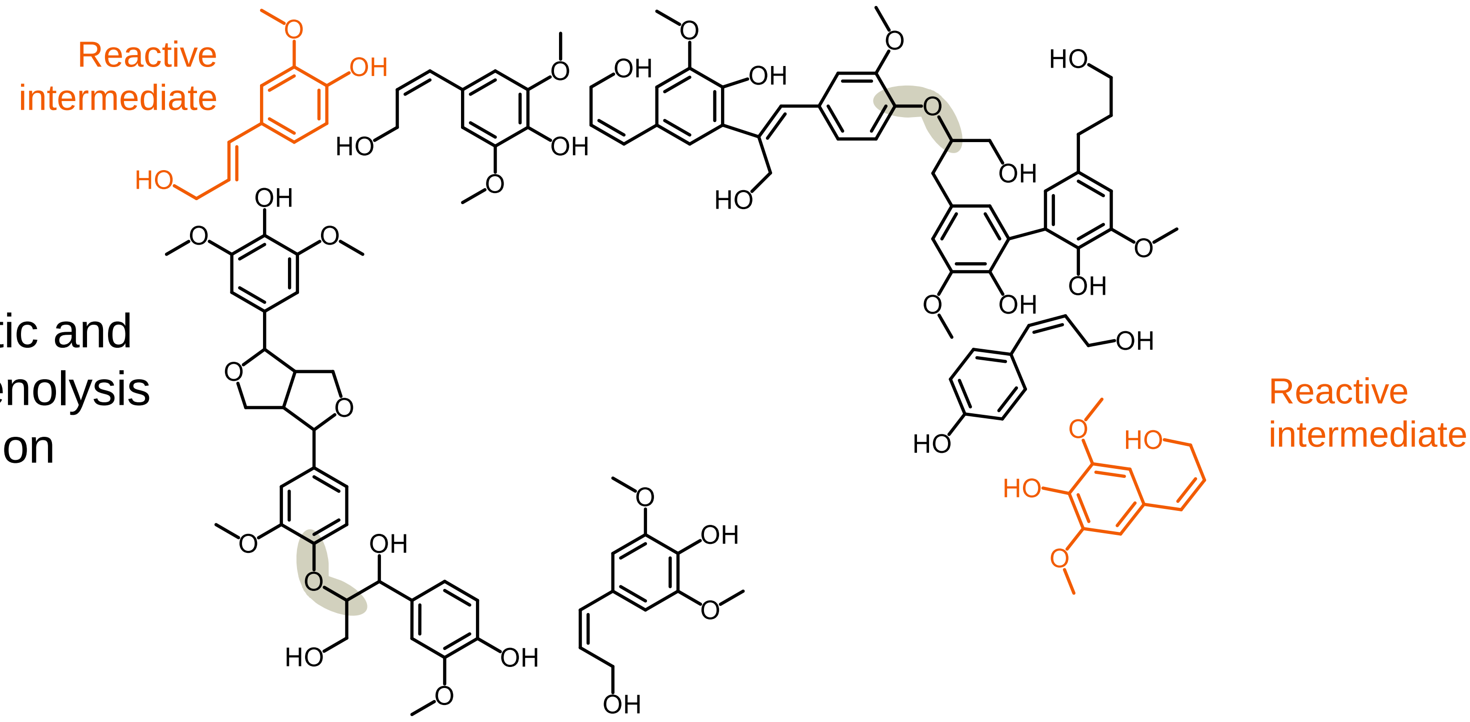
THE CHEMISTRY OF RCF …



Renders *et al. Energy Environ. Sci.*, **2017**

... BY CHEMICALLY STABILIZING REACTIVE INTERMEDIATES

Solvolytic and hydrogenolysis action



Renders *et al.* *Energy Environ. Sci.*, **2017**

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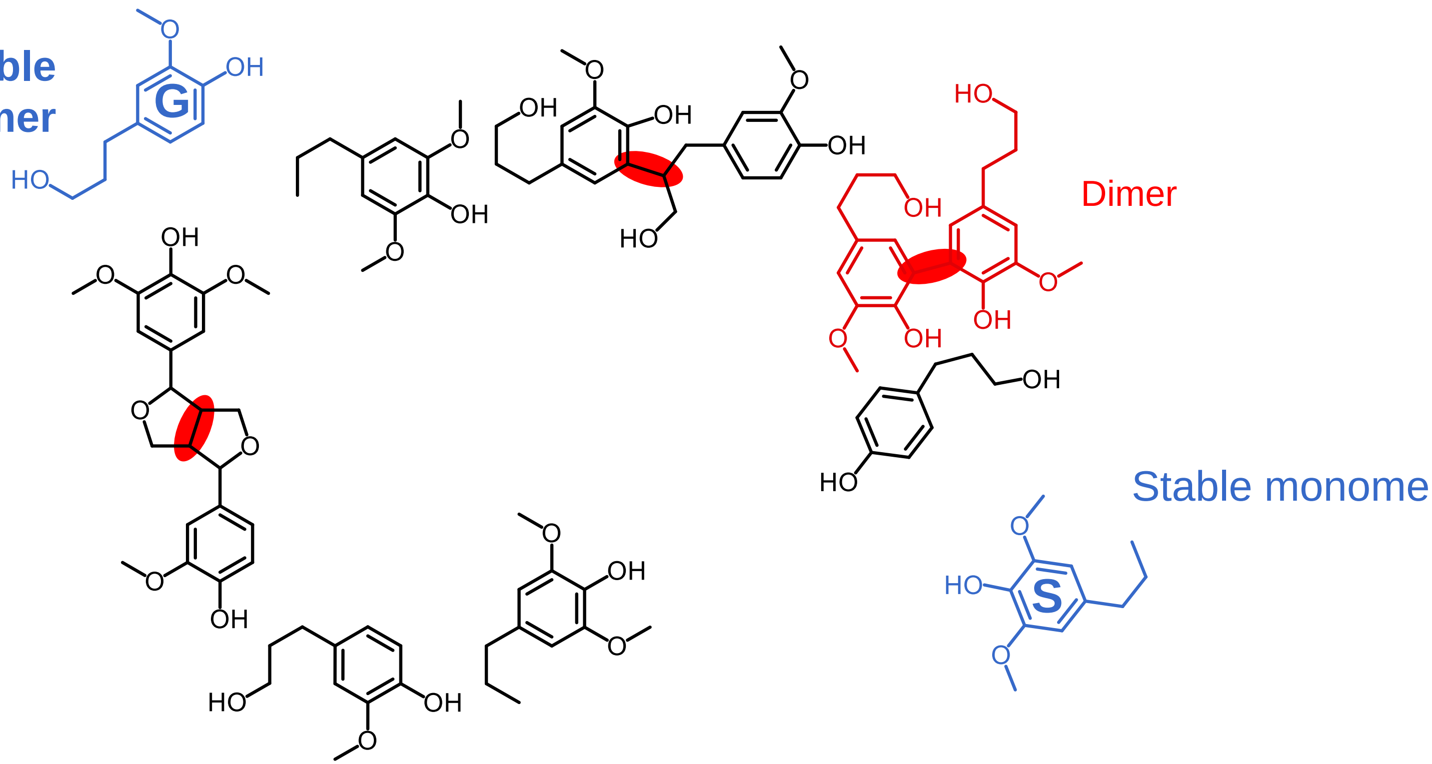
… INTO STABLE PHENOLIC PRODUCTS



Catalytic hydrogenation action

Renders *et al. Energy Environ. Sci.*, **2017**

Lignin-first **RCF biorefinery and the role of catalysis**

Renders *et al.* Current opinion in biotechnology, 2019

BENCH SCALE RCF BIOREFINERY:
Lignin oil - Pulp

Delignification yield: > 90%

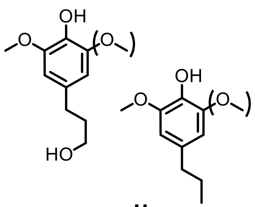
Lignocellulose sawdust
e.g. poplar, birch, pine, eucalyptus

Solvent
e.g. methanol

Catalyst + H_2
e.g. Pd/C, Ru/C, Ni/SiO_2

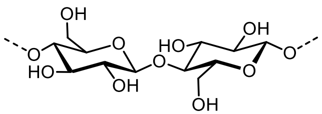
Batch reactor (100 mL)
200 - 250 °C
60 - 120 bar

Lignin oil



+ dimers
oligomers

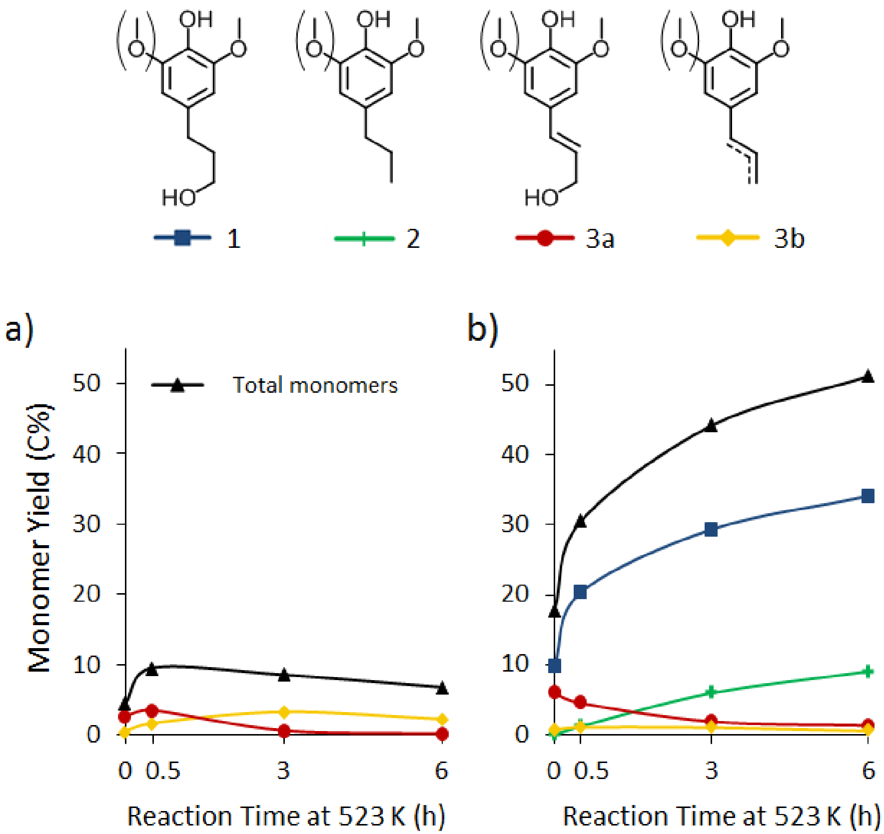
Pulp (+ catalyst)
Cellulose
Hemicellulose



Van den Bosch and Schutyser *et al. Energy Environ. Sci.*, **2015**
Renders *et al. ACS Catalysis*, **2016;**
Renders *et al. ACS Sustainable Chemistry & Engineering.* **2016**
Renders *et al.* *Energy Environ. Sci.*, **2017**
Renders *et al*. *Green Chemistry* **2018**

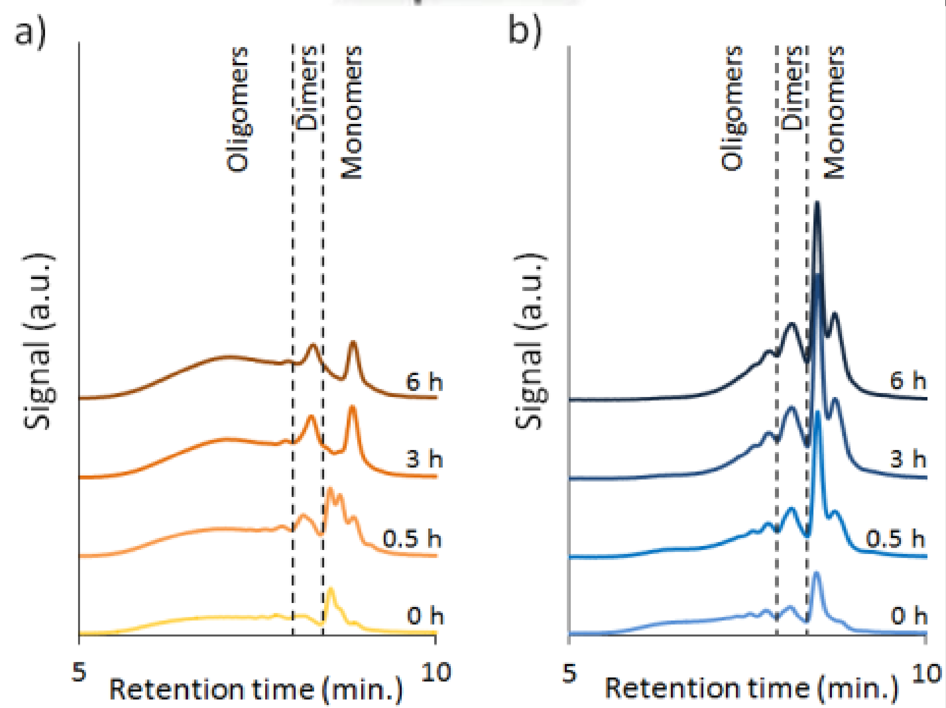
Pulp retention: > 95% Hexoses
> 85% Pentoses

ROLE OF CATALYSIS:
1. HIGH LIGNIN MONOMER YIELD



Without Catalyst

With Catalyst



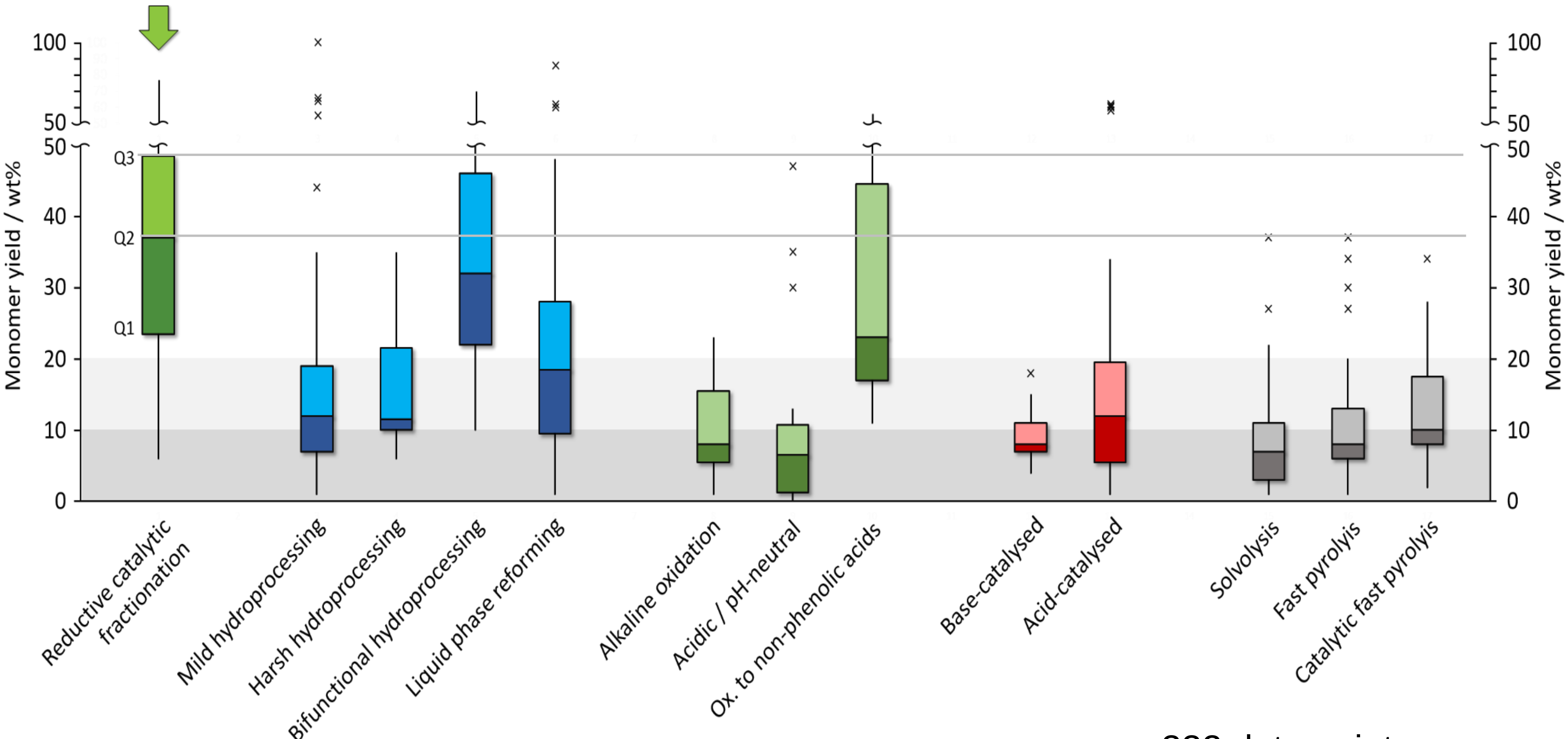
Without Catalyst

With Catalyst

Van den Bosch *et al*. Green Chemistry, 2017.

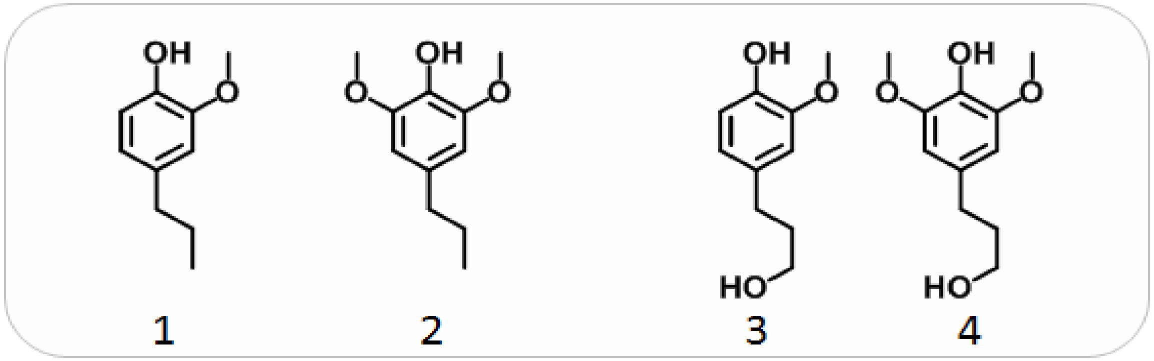
MONOMER YIELD IN OTHER BIOREFINERIES

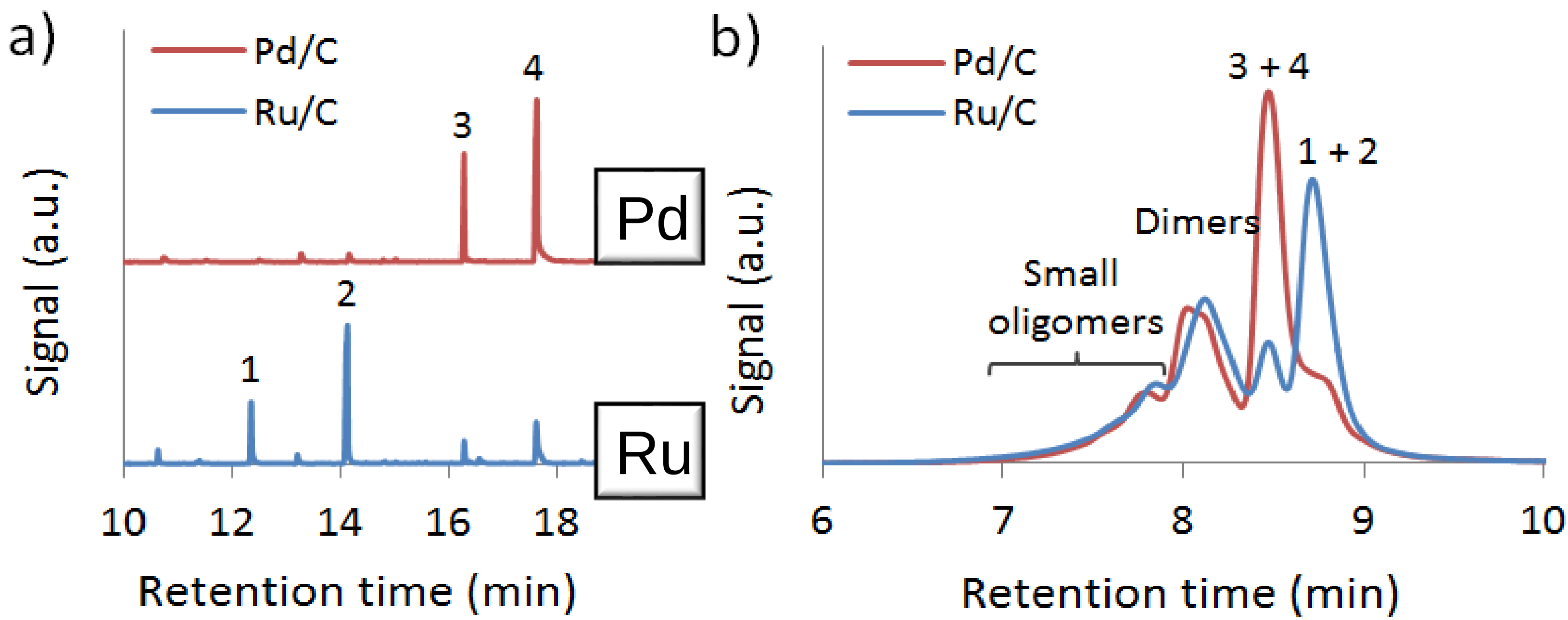
63 data points
26 studies



332 data points
170 studies

ROLE OF CATALYSIS:
2. CONTROL PRODUCT SELECTIVITY





Schutyser et al. Chem Commun **2015,** 51, 131

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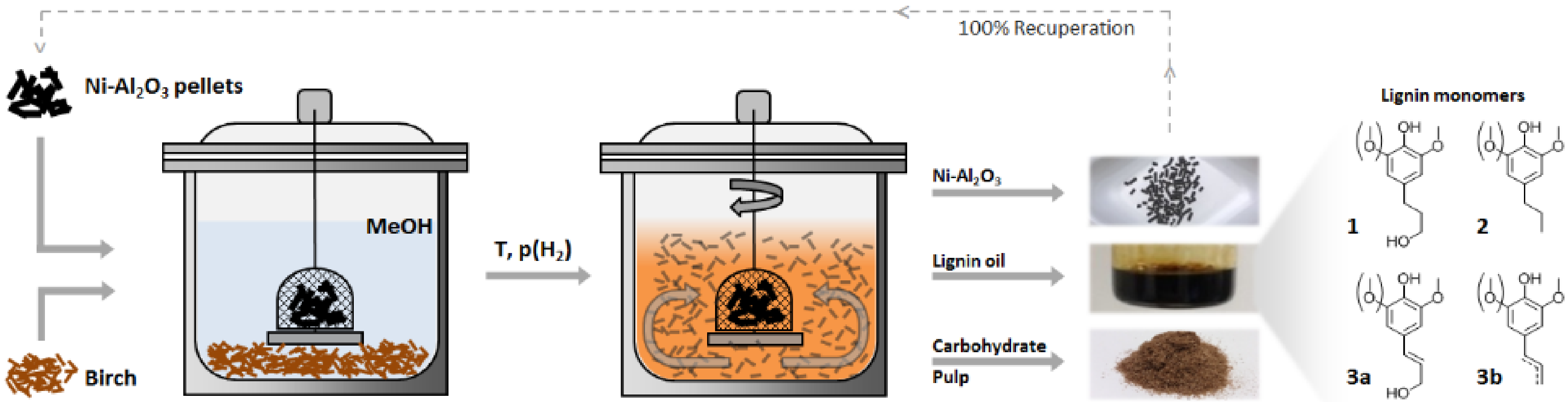
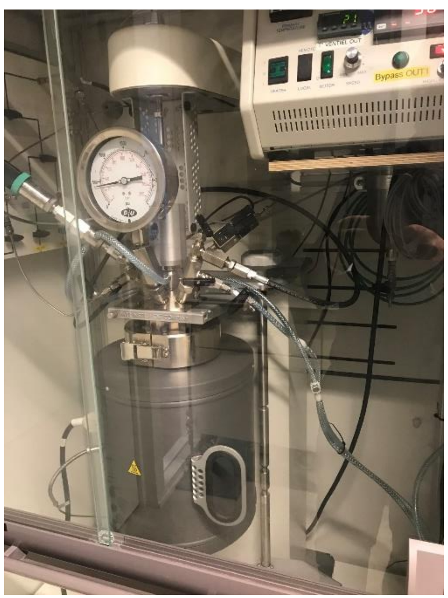
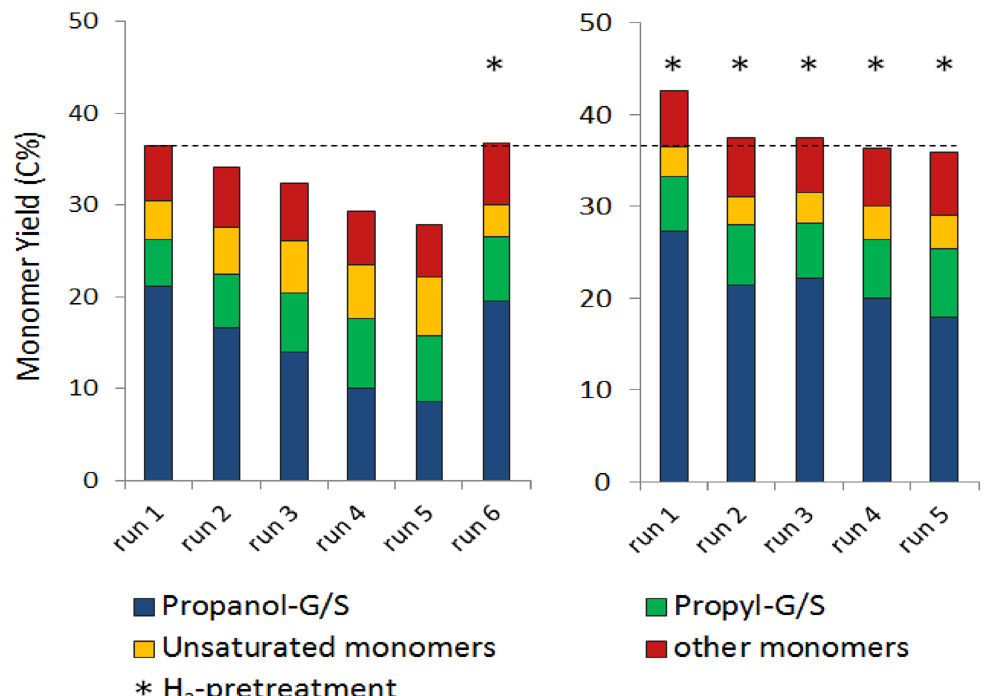
PRACTICAL CATALYST USAGE AND RECYCLE





2 L facility



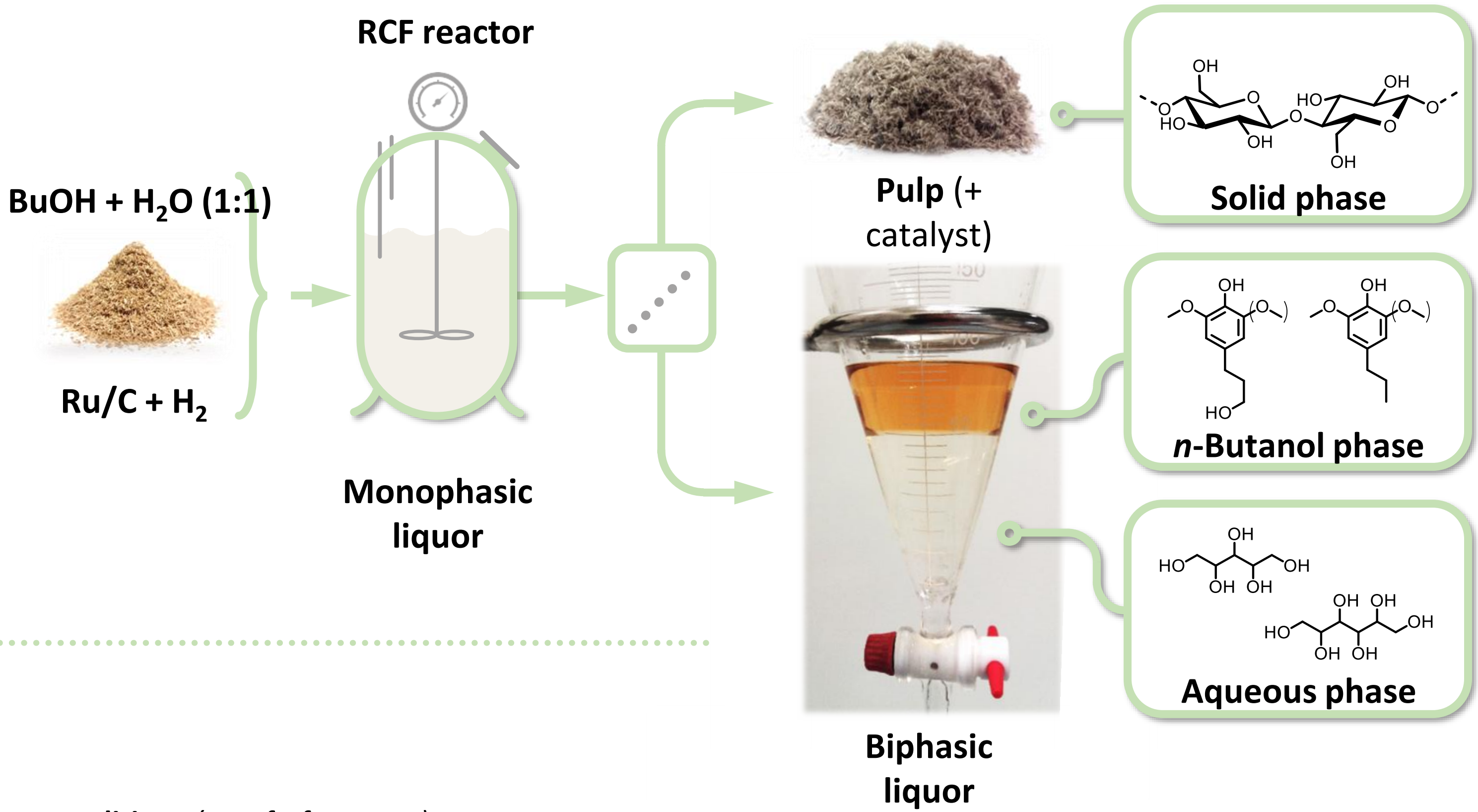
Van den Bosch *et al*. Green Chemistry,

Upscaling the RCF biorefinery at KULeuven

BIOCON®

IMPACT OF SCALING ON PRODUCT PROPERTIES

One phase MeOH
Two phase BuOH-water



+ oligomers

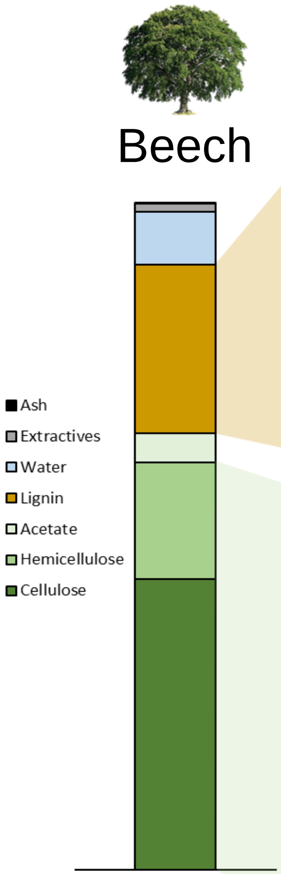
Process conditions *(proof-of-concept)*

200 °C 2 g eucalyptus sawdust
10 bar H_2 0.1 g Ru/C (5 wt% metal)
2 h 20 mL *n*-butanol + 20 mL H_2O

Renders *et al.* GC 2018

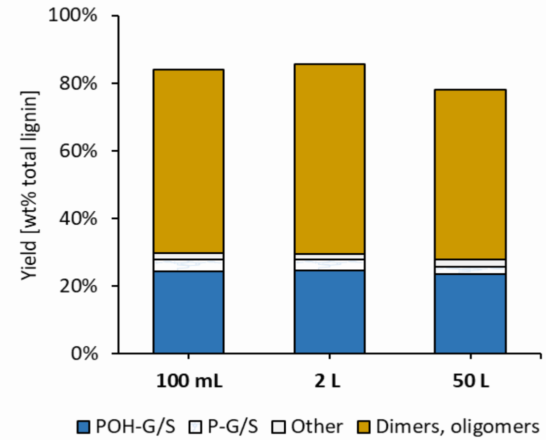
IMPACT OF SCALING ON THE PRODUCT PROPERTIES

BIOCON® Fraunhofer CLIB KATHOLIEKE UNIVERSITEIT LEUVEN



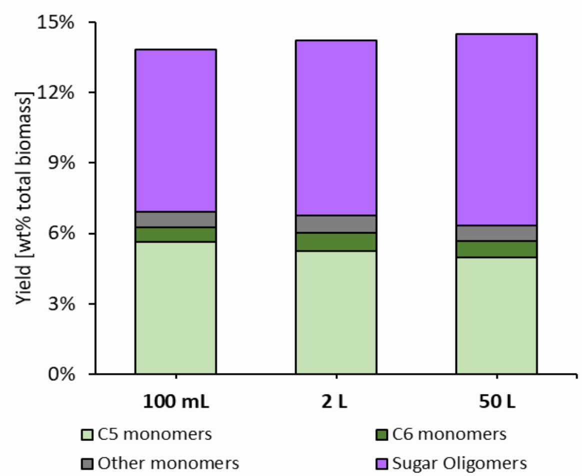
Ru/C
200°C, 2h, 30 bar H_2
40 V% (n-butanol/water)
40 g/L biomass, 4 g/L catalyst

A Lignin oil



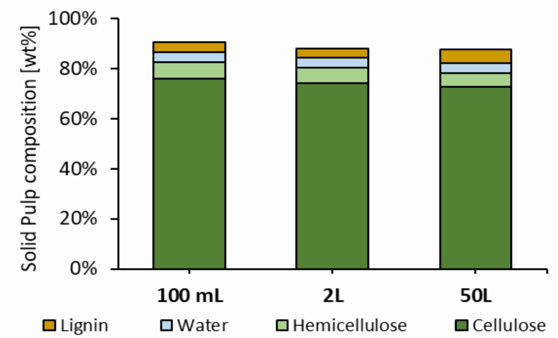
	100 mL	2L	50L
Delignification [wt% total lignin]	84	85	78
Monomer yield [wt% oil]	36	34	36
Mn	603	589	571
Mw	895	970	769
PDI	1,48	1,65	1,35
S_{BO4} [%]	1,2	1,2	1,5
S_{POH} [%]	82	84	85

B Soluble sugar products



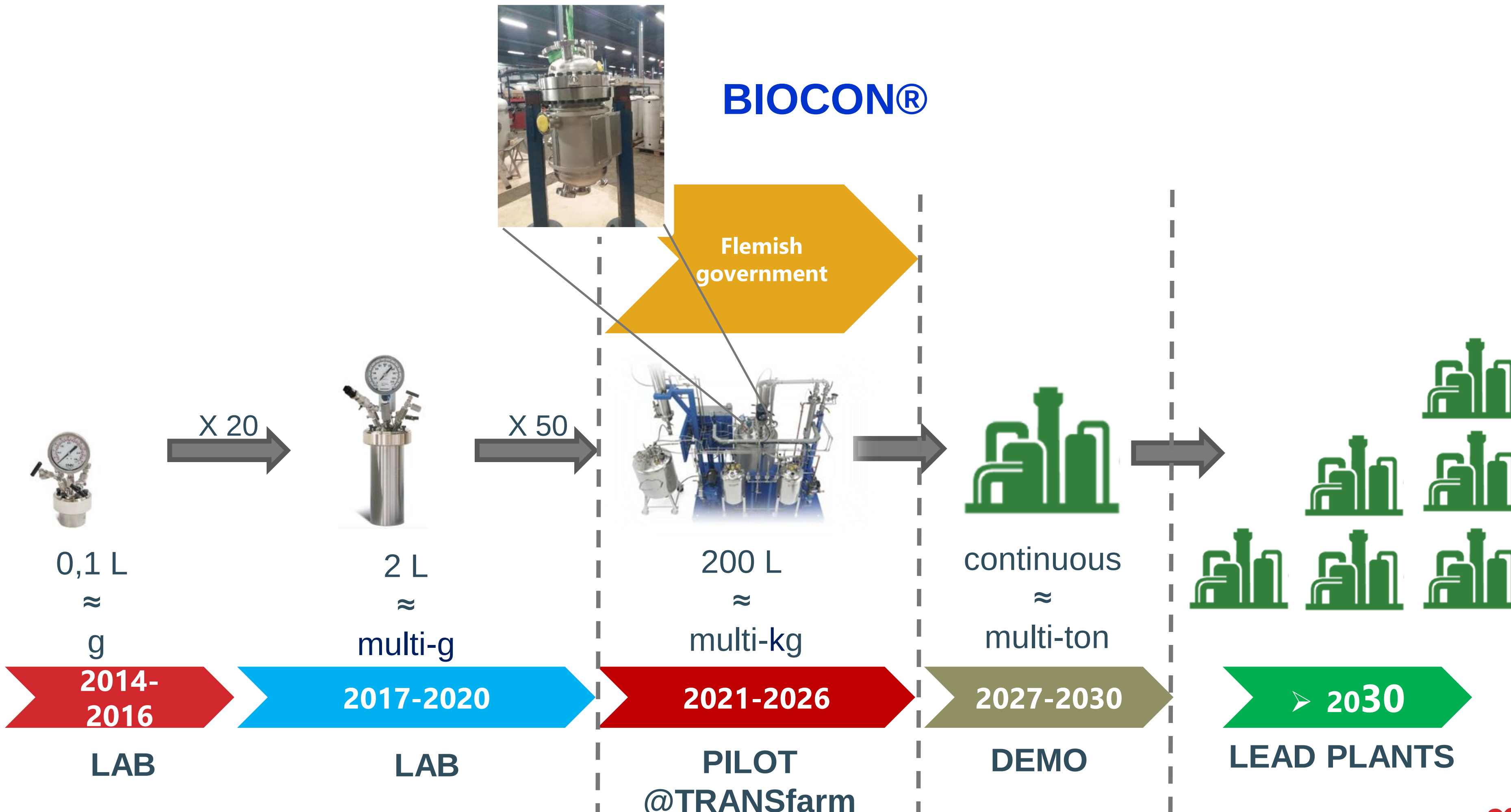
C Solid pulp

	100 mL	2L	50L
Pulp retention (wt%)	51	50	52
Sacharification (%)	93	84	74
Fermentation efficiency (%)	85	83	100

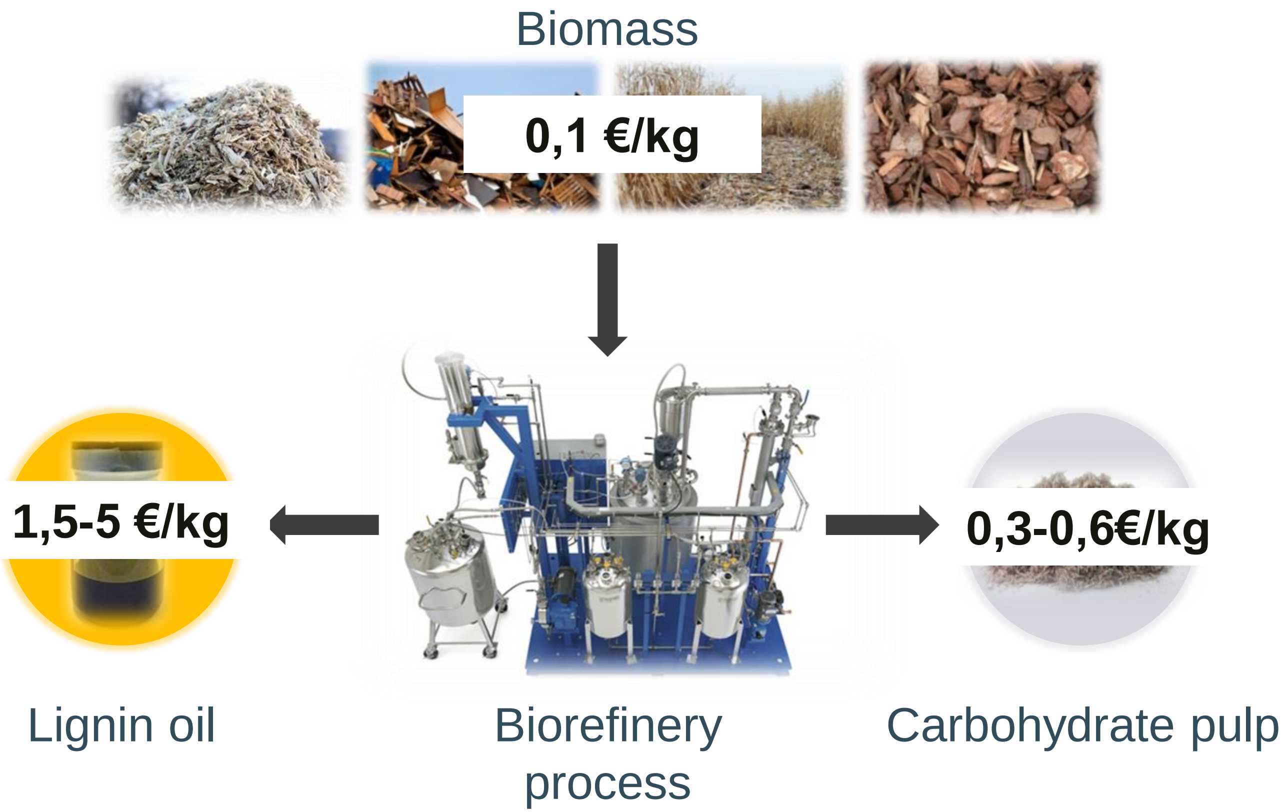


Cooreman *et al.* in preparation

ROADMAP LIGNIN BIOREFINERY@KULEUVEN: BIOCON®

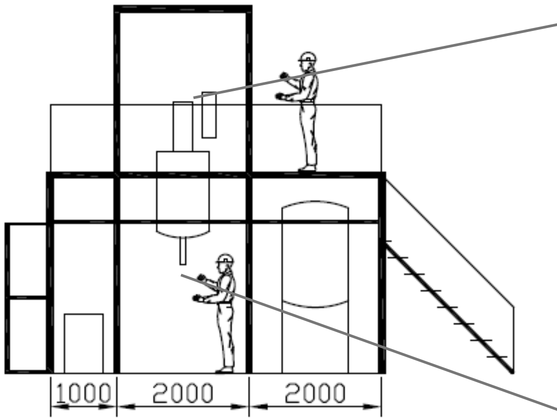


BIOREFINERY TECHNOLOGY AT LARGE SCALE: TARGETS



BIOCON®

BIOCON BIOREFINERY PILOT ON TRANSFARM SITE



Funded by the European Union
NextGenerationEU

DE VLAAMSE VEERKRACHT

Horizon 2020
European Union Funding for Research & Innovation

BBI JU

Bio-based Industries Consortium

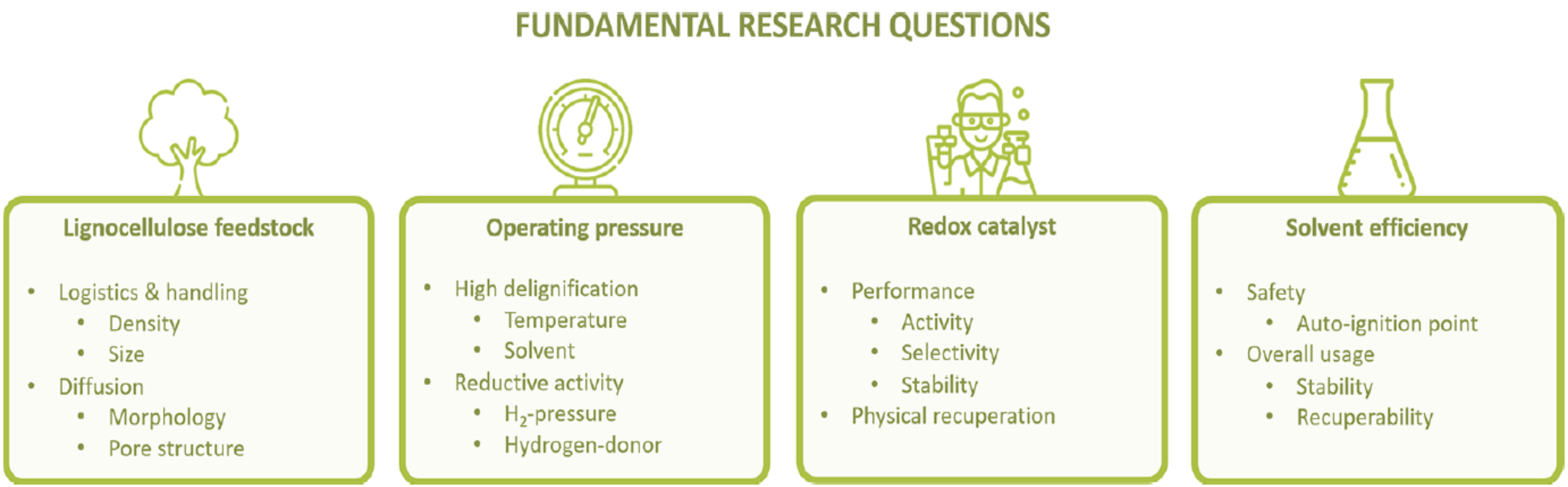
SMARTBOX

Identification of challenges in the BIOCON® program

- **fundamental questions (conditions, catalyst, solvent)**
- **reactor design**
- **process: TEA & LCA**

CHALLENGES: FUNDAMENTAL QUESTIONS

BIOCON®



Cooreman E. *et al*. Ind Eng Chem Res. 20

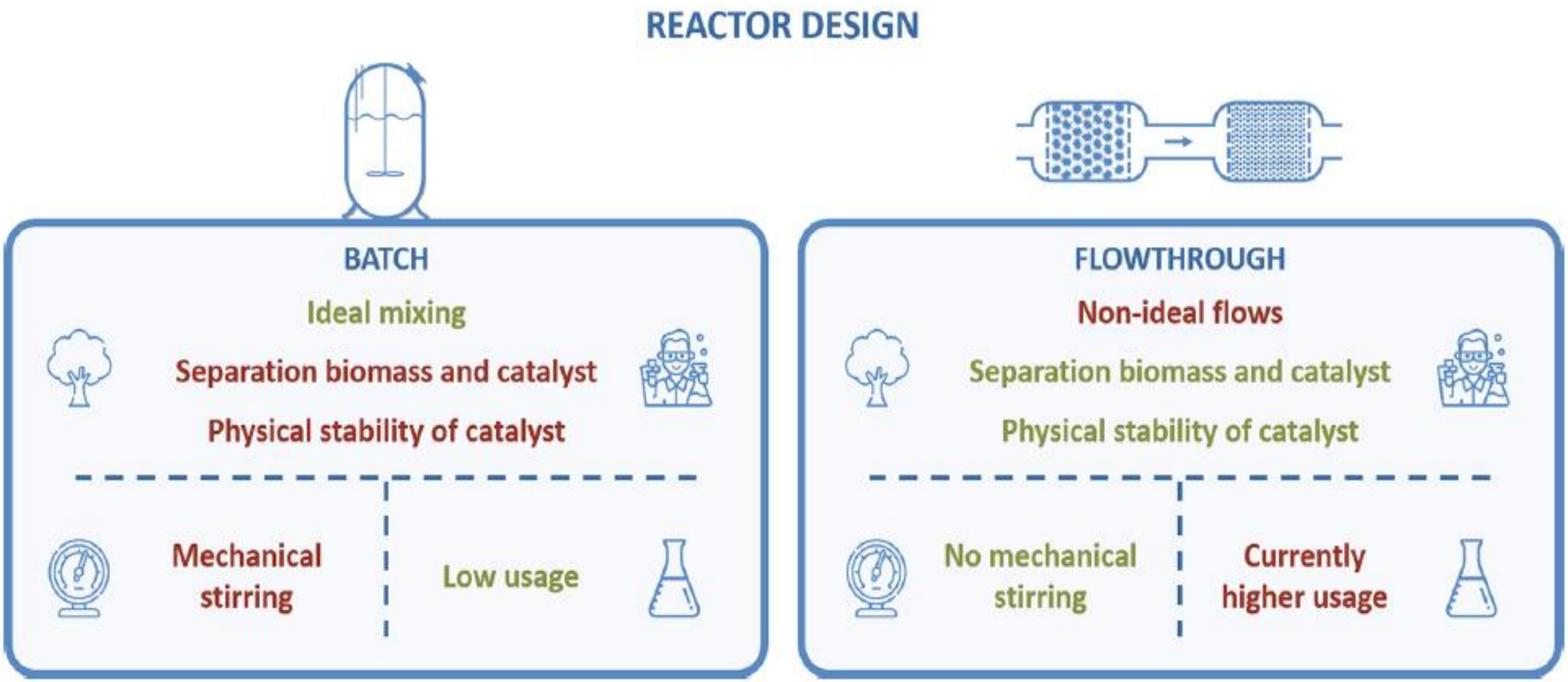
Bark ?
(tannines, suberines)

Vangeel T *et al*. Biomass conversion & Biorefinery, 20
Vangeel T *et al*. Green Chemistry, 2019

Waste woods ?
(Wood variability, metal and oxide impurities, …)

Van den Bossch G. *et al*. submitted

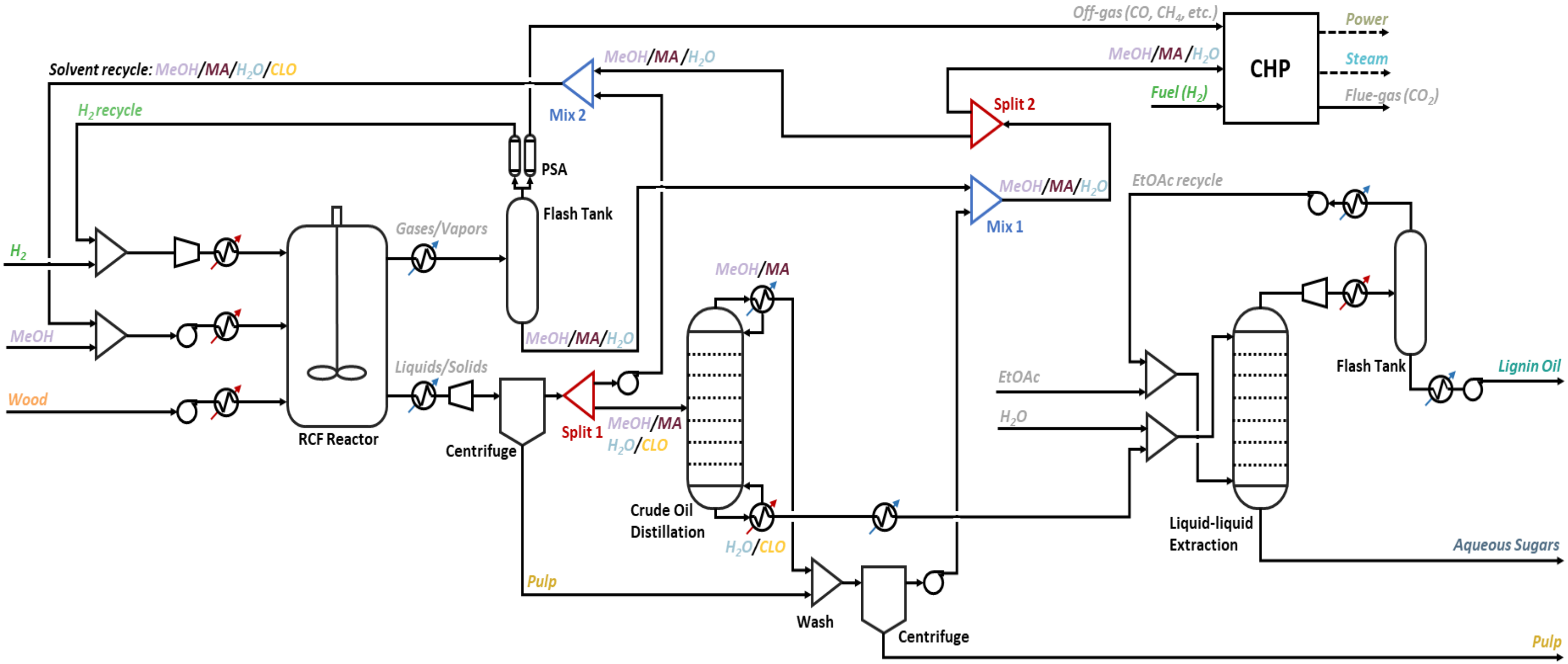
CHALLENGES: REACTOR DESIGN



Cooreman E. *et al*. Ind Eng Chem Res. 20

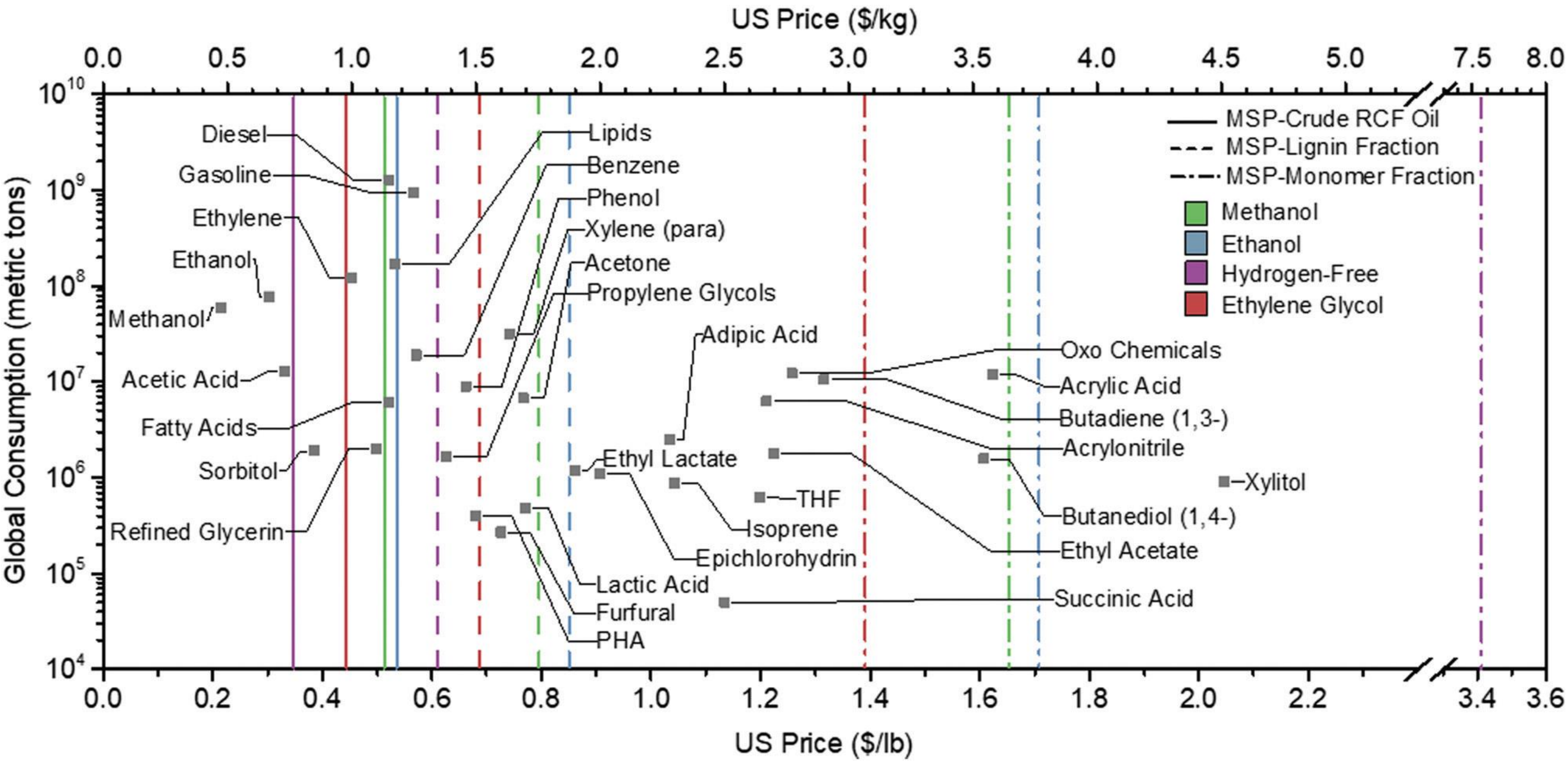
PROCESS CHALLENGES : TEA & LCA

BIOCON®



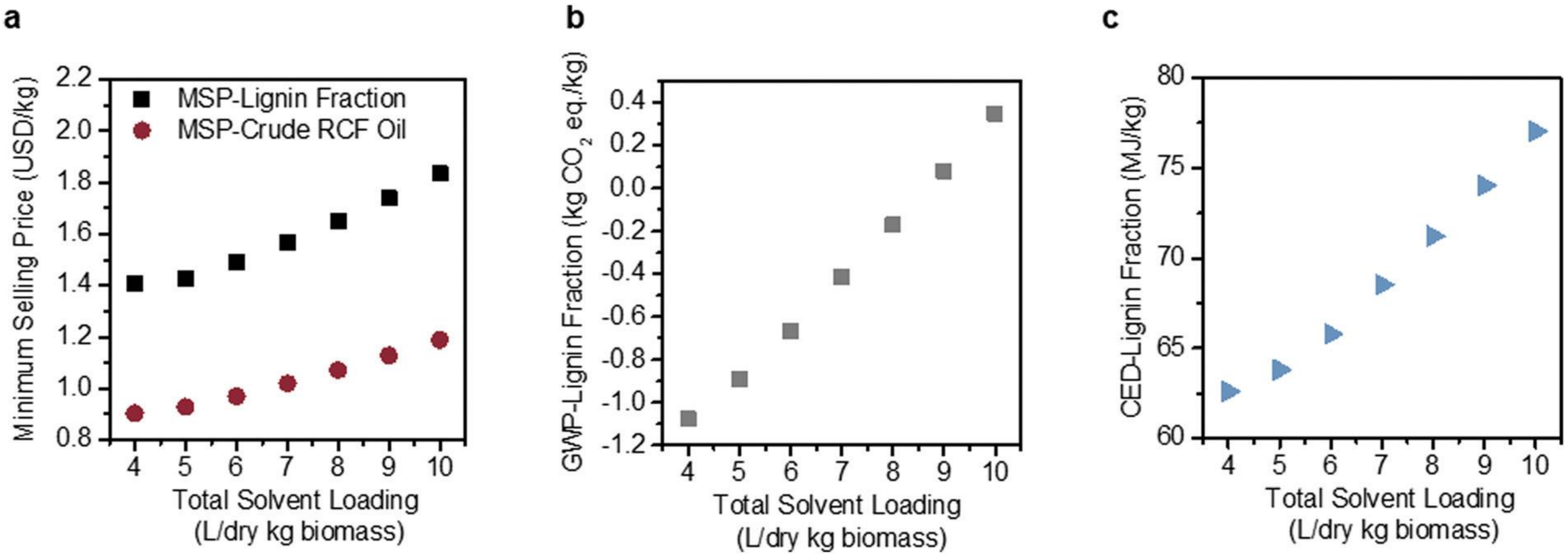
Bartling et al. EES, 2021
Arts W. *et al*. (in preparation)

ECONOMICS



Bartling et al. EES, 2021

CO2 FOOTPRINT & ECONOMICS



Bartling et al. EES, 2021

Holistic view on the RCF biorefinery and usage of the primary products

lignin oil & *pulp*:

PULP USAGES

Fibre composites
Insulation

Paper
Cardboard

Platform
chemicals

Food
additives

Biofuels

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LIGNIN OIL

Resins
PURs
Epoxys

SOUDAL
LAWTER™
RECTICEL insulation
tfc BIOMASS BASED CHEMICALS
Henkel

CHEMSTREAM SUSTAINABLE CHEMISTRY
Beaulieu International Group

Flameretardents
Antioxidants
Plasticizers
Emulsifiers
…

Aromatic Polymers

Functional additives

covestro

Biolubricants

oleon

(di)Phenol alkylphenols

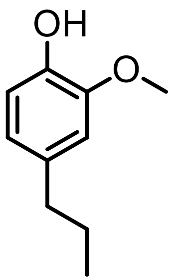
Bioactive compounds

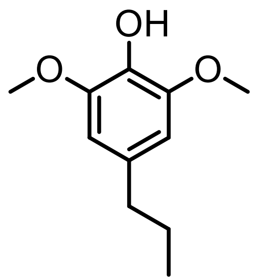
HUVEPHARMA
nuscience safe & innovative nutrition

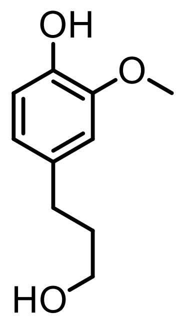
Phenols
Bisphenols

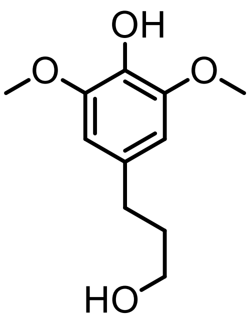
SOLVAY
INEOS
sabic
EASTMAN
KelAda

Examples of applications with monomers

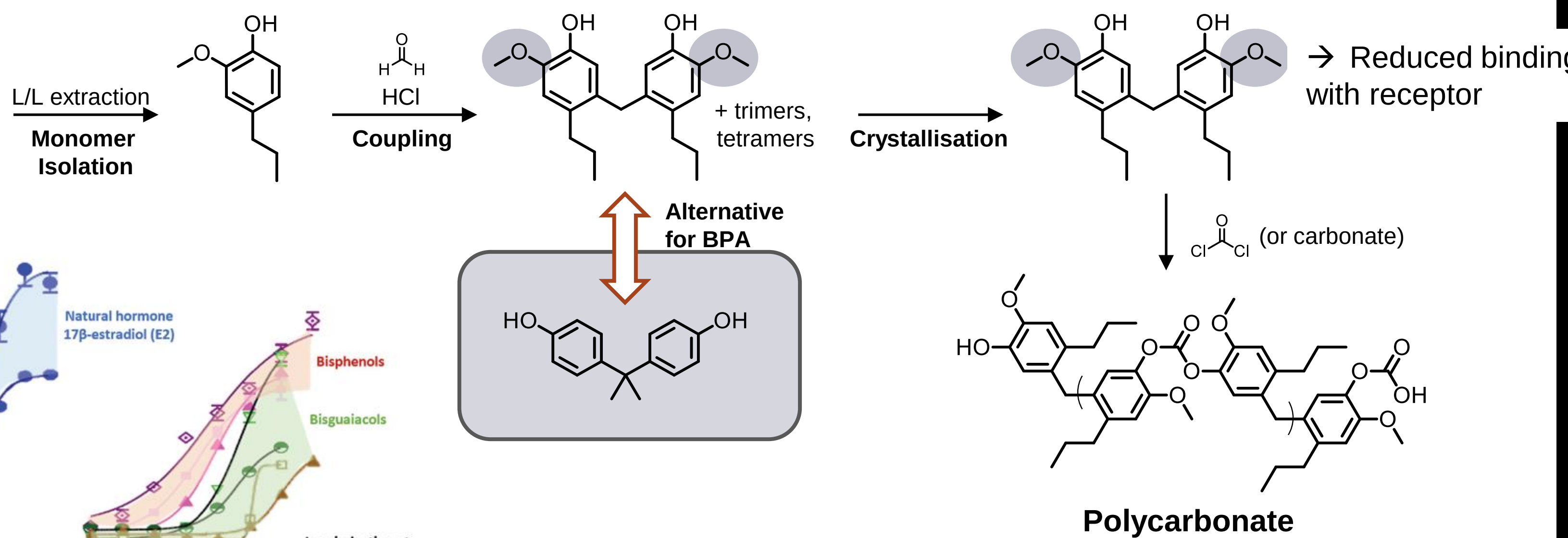








New chemical example 1: bisphenols



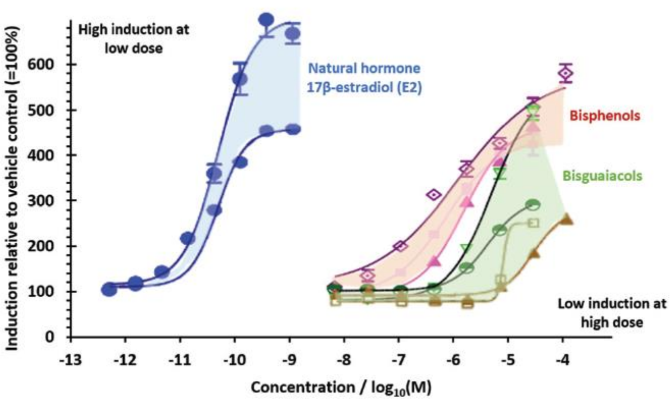


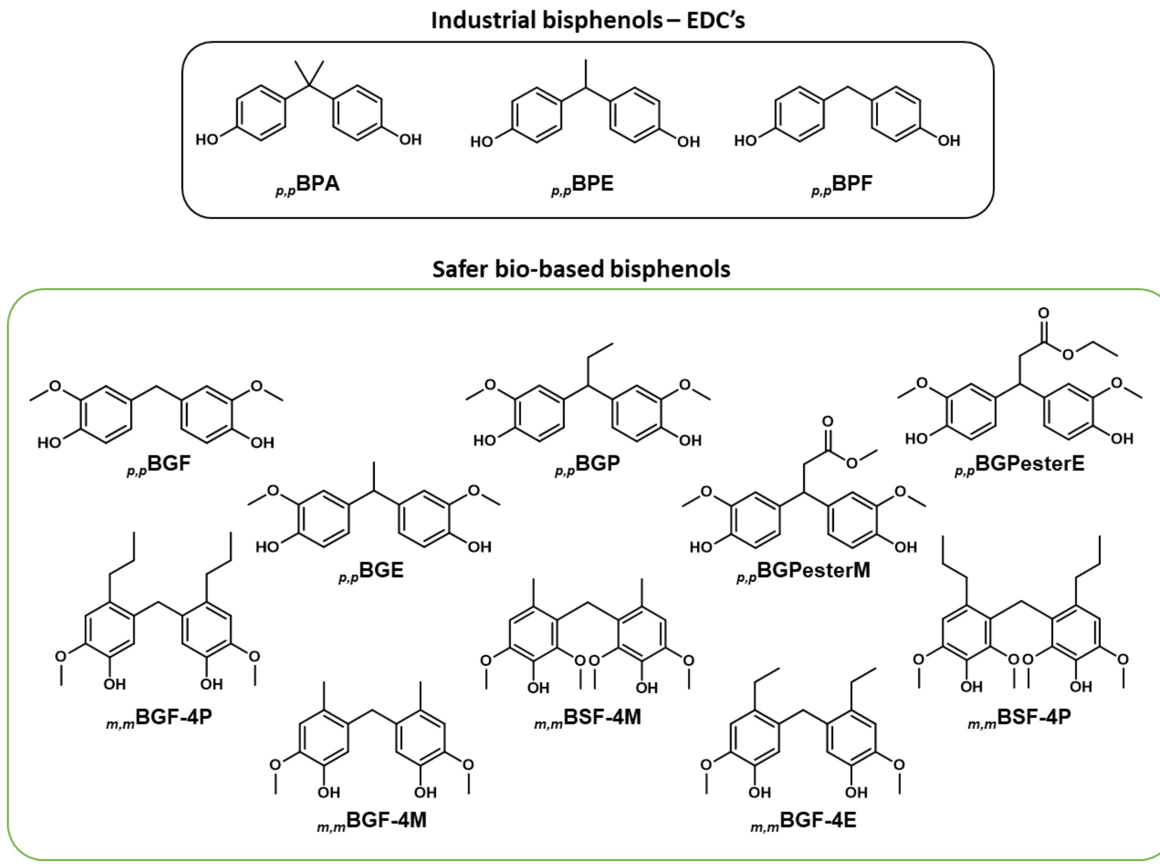
Fig. 3 *In vitro* screening of the estrogenic potency of bis(4-alkylguaiacol)s *via* an estrogen-responsive reporter assay in human MCF-7 cells (MELN assay). Three zones of sigmoidal responsiveness are indicated for reference estrogen (blue), commercial bisphenols (magenta) and bisguaiacols (green). The individual test compounds: E2 (blue, ●), BPF (dark mag., ◇), BPA (light mag., ■), BPE (mag., ▲), 4-methyl- (light green, ▽), 4-ethyl- (dark green, ◓), 4-*n*-propyl- (light brown, □) and 5-methyl- (dark brown, ▲) bisguaiacols.

For example:
Polycarbonate plexiglass
Covestro (Antwerp)

Patented technology BIOCON®

Koelewijn, S.-F. *et al. Green. Chem.* 19, 2561-2570 (20
Koelewijn, S.-F. *et al. Green. Chem.* 20, 1050-1058 (20
Trulleman *et al. Polymer Chemistry* 2021 (online)

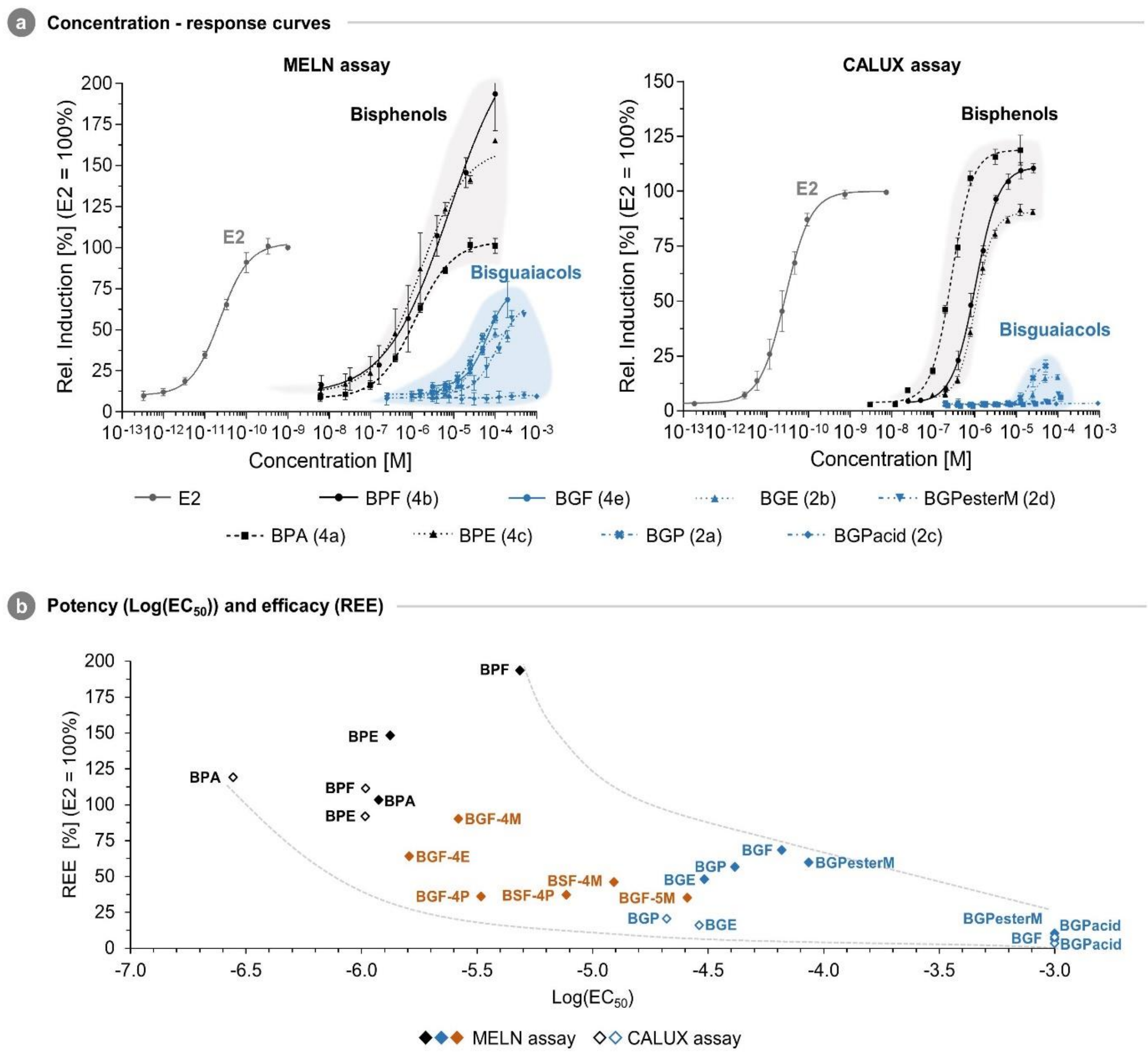
New chemical example 1: safer bisphenols platform



Bisphenol platform

Trullemans et al. Polymer 2021

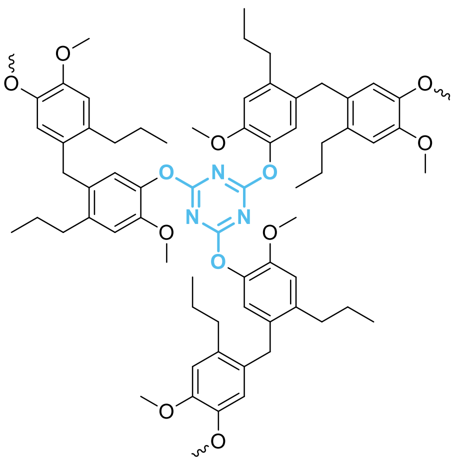
NEW CHEMICAL EXAMPLE 1: SAFER BISPHENOLS PLATFORM



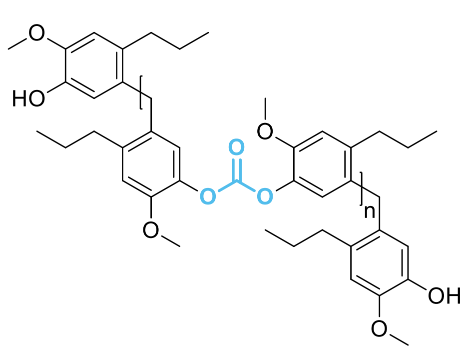
Trullemans & Koelewijn et al. submitted

New chemical example 1: bisphenols

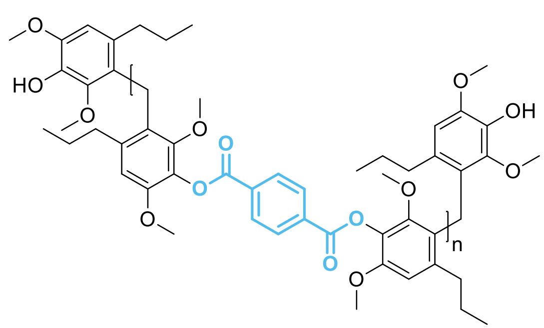
cyanate ester resins



polycarbonates



polyesters

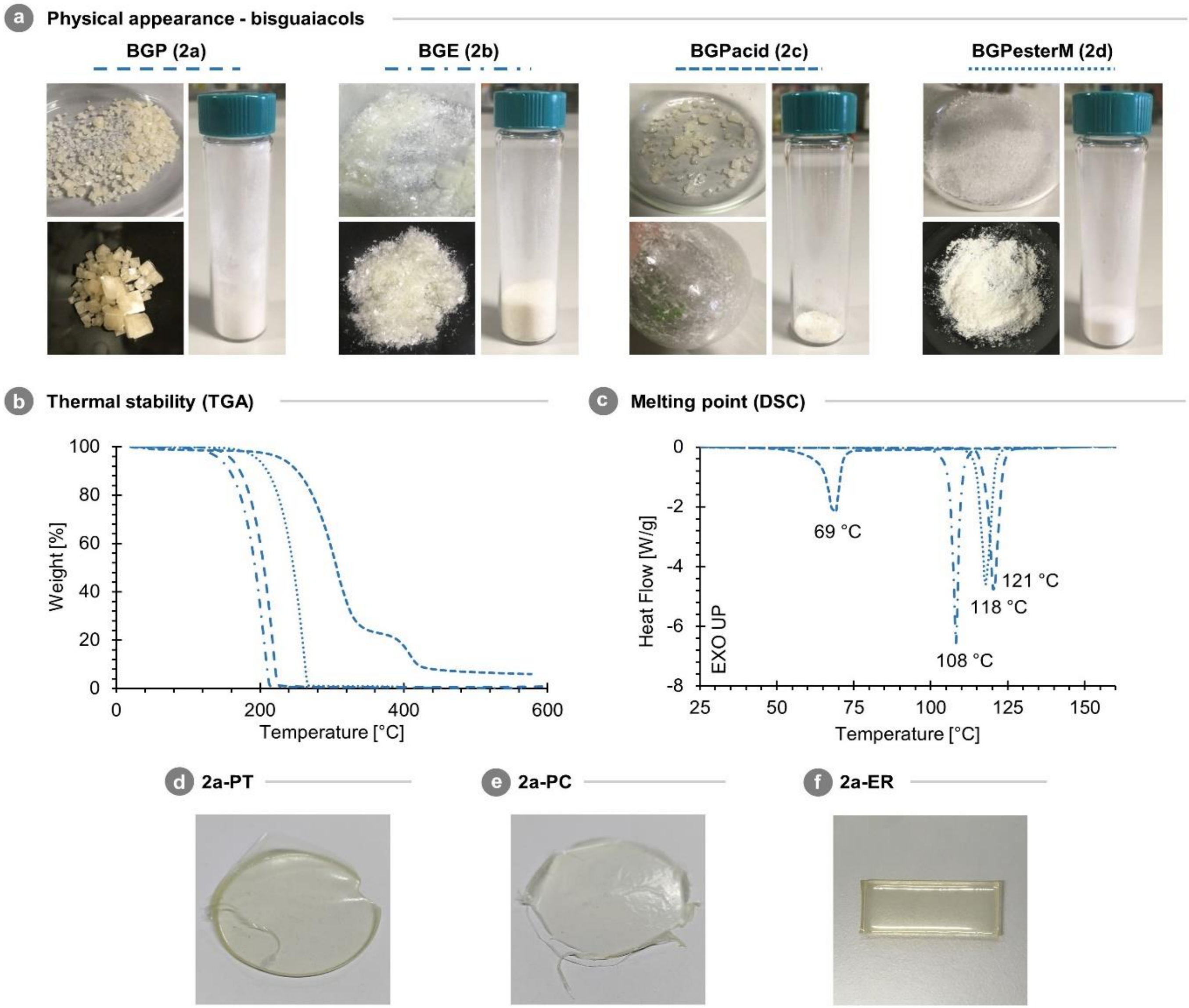


Patented technology BIOCON®

Koelewijn, S.-F. *et al. Green. Chem.* **19**, 2561-2570 (201

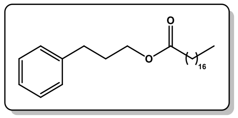
Koelewijn, S.-F. *et al. Green. Chem.* **20**, 1050-1058 (201

New chemical example 1: bisphenols

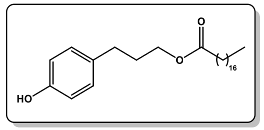


Patented technology BIOCON®

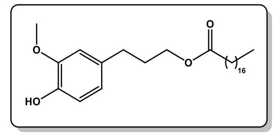
NEW CHEMICAL EXAMPLE 2: GUAIACOL AND SYRINGOL ESTERS - PLASTICIZERS



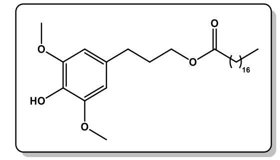
PDL1
M.W. = 402,66
Yield: 68%



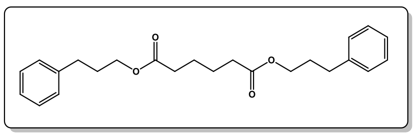
PDL4
M.W. = 418,66
Yield: 72%



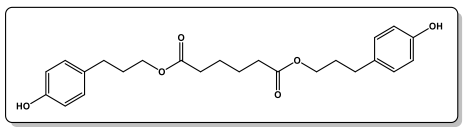
PDL5
M.W. = 448,69
Yield: 76%



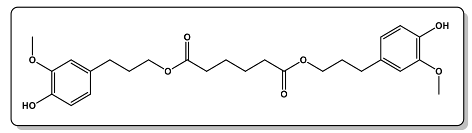
PDL6
M.W. = 478,71
Yield: 50%



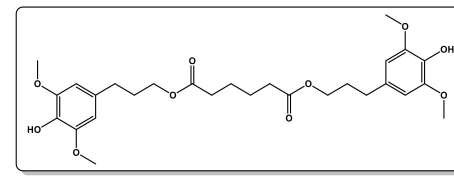
PDL7
M.W. = 382,50
Yield: 77%



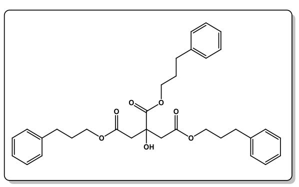
PDL8
M.W. = 414,50
Yield: 88%



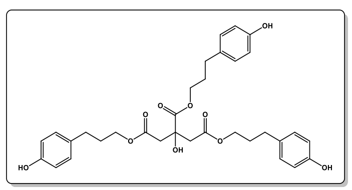
PDL9
M.W. = 474,55
Yield: 83%



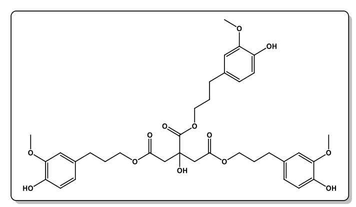
PDL10
M.W. = 534,60
Yield: 44%



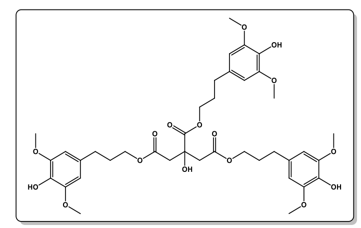
PDL11
M.W. = 546,66
Yield: 80%



PDL12
M.W. = 594,66
Yield: 88%



PDL13
M.W. = 684,74
Yield: 68%

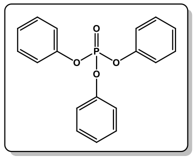


PDL14
M.W. = 774,81
Yield: 57%

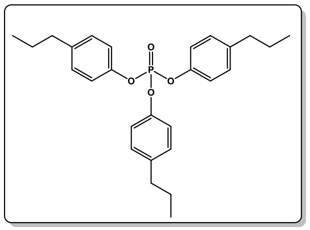
Submitted data

10 to 100 g scale

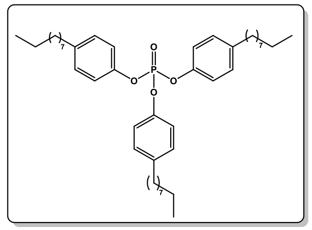
NEW CHEMICAL EXAMPLE 2: GUAIACOL AND SYRINGOL PHOSPHATES - FLAMERETARDENTS



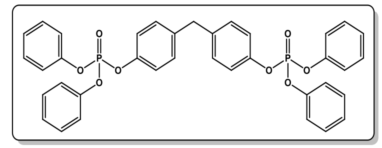
P3OP
Commercial sample



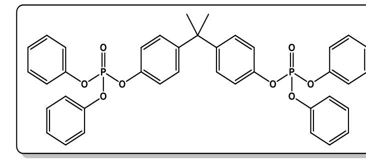
JN002-9
Yield: 51%



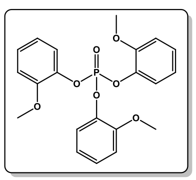
JN011
Yield: 71%



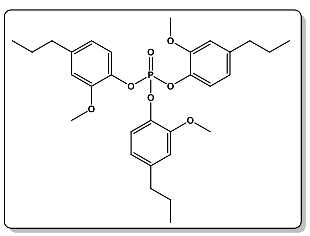
SCD314
Yield: 96%



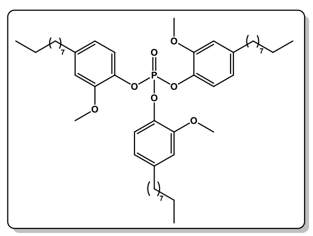
SCD315
Yield: 94%



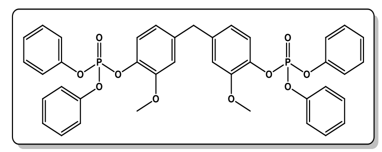
G3OP
Yield: 92%



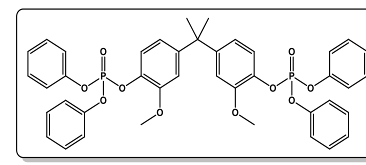
SCD296
Yield: 91%



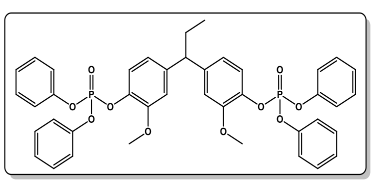
JN022
Yield: 32%



SCD316
Yield: 93%



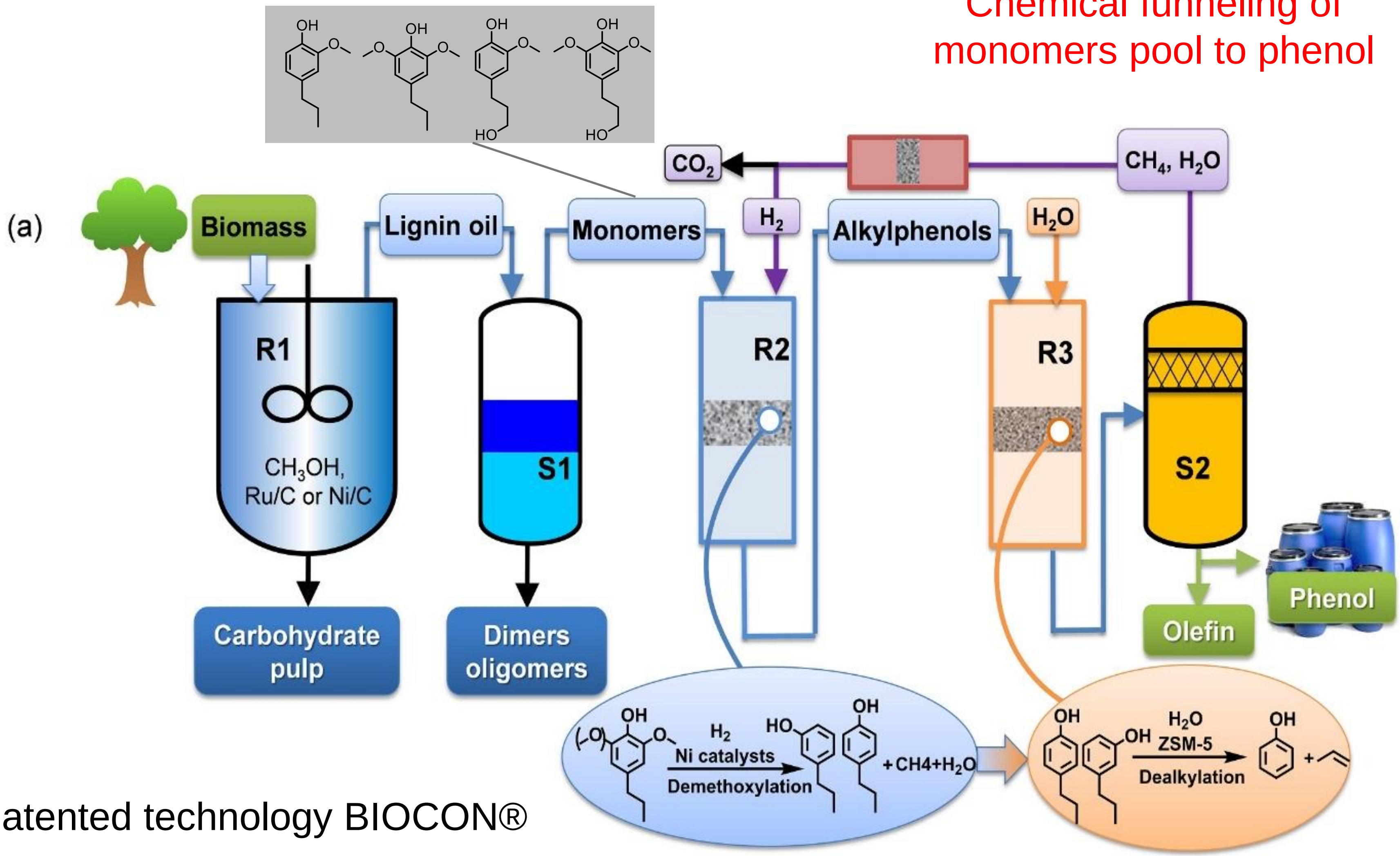
JN007
Yield: 80%



JN003
Yield: 83%

Drop-in chemicals example 2: Phenol

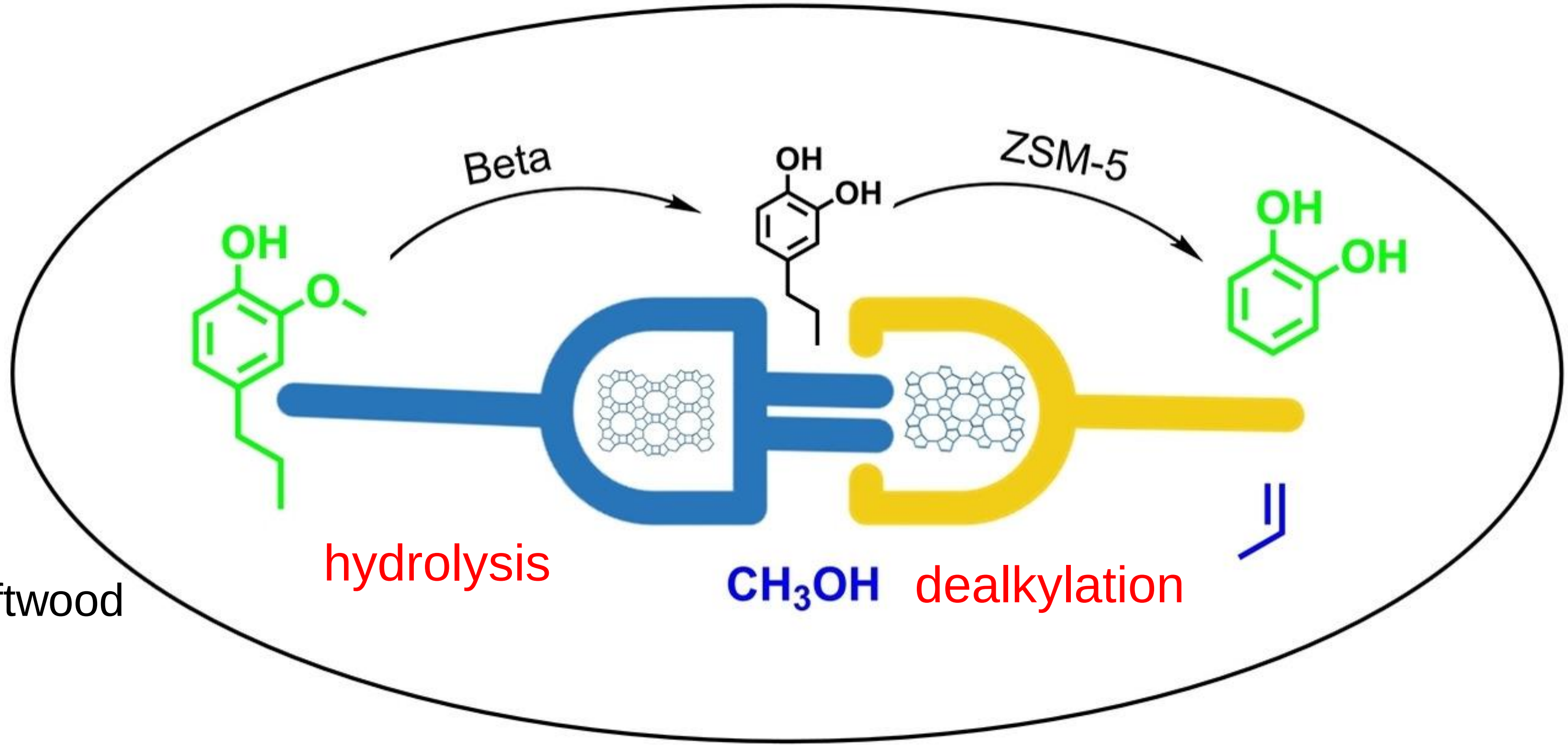
Chemical funneling of monomers pool to phenol



Patented technology BIOCON®

Verboekend and Liao et al. *Green Chem*. 2016; Liao et al. *ACS Catalysis* 2018; Liao et al. *Appl Catal B Env*. 2018; *ACS Sust&Eng* 2020; Liao et al. *Science* 2020;

Drop-in chemicals example 3: Catechol



softwood

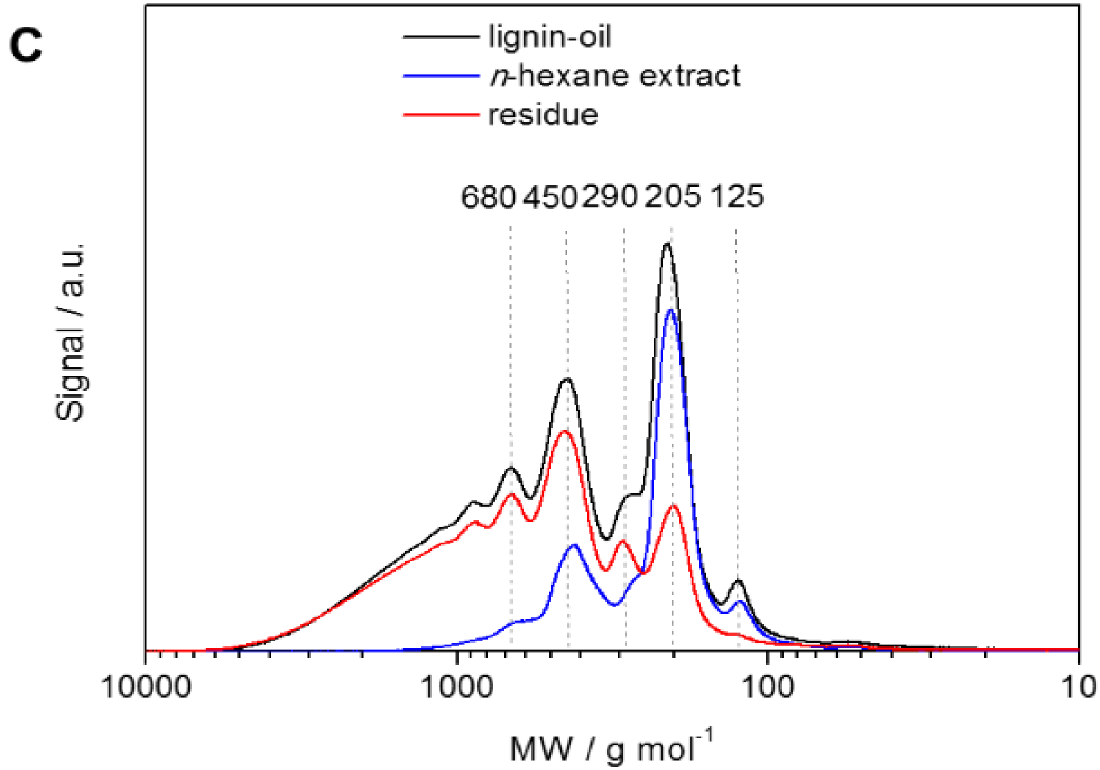
Wu X. et al. *ChemSusChem* 2022

What about utilizing the oligomers ?

molecular understanding of the chemistry

design towards applications

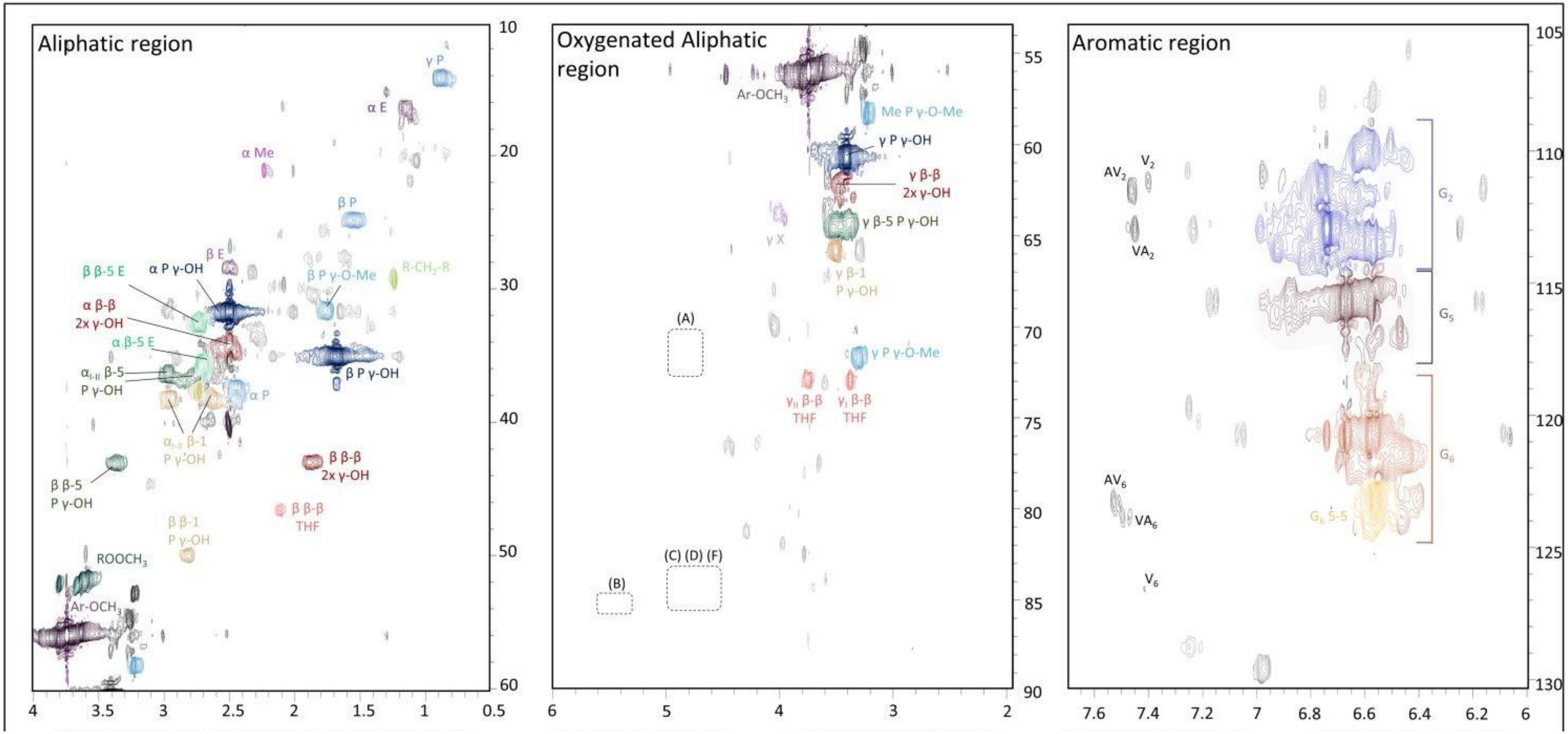
MOLECULAR WEIGHT DISTRIBUTION USING GPC



M_W
M_n
D

Liao and Koelewijn *et al*., Science 202

MOLECULAR INSIGHT INTO THE OLIGOMER STRUCTURE : 2D HSQC NMR

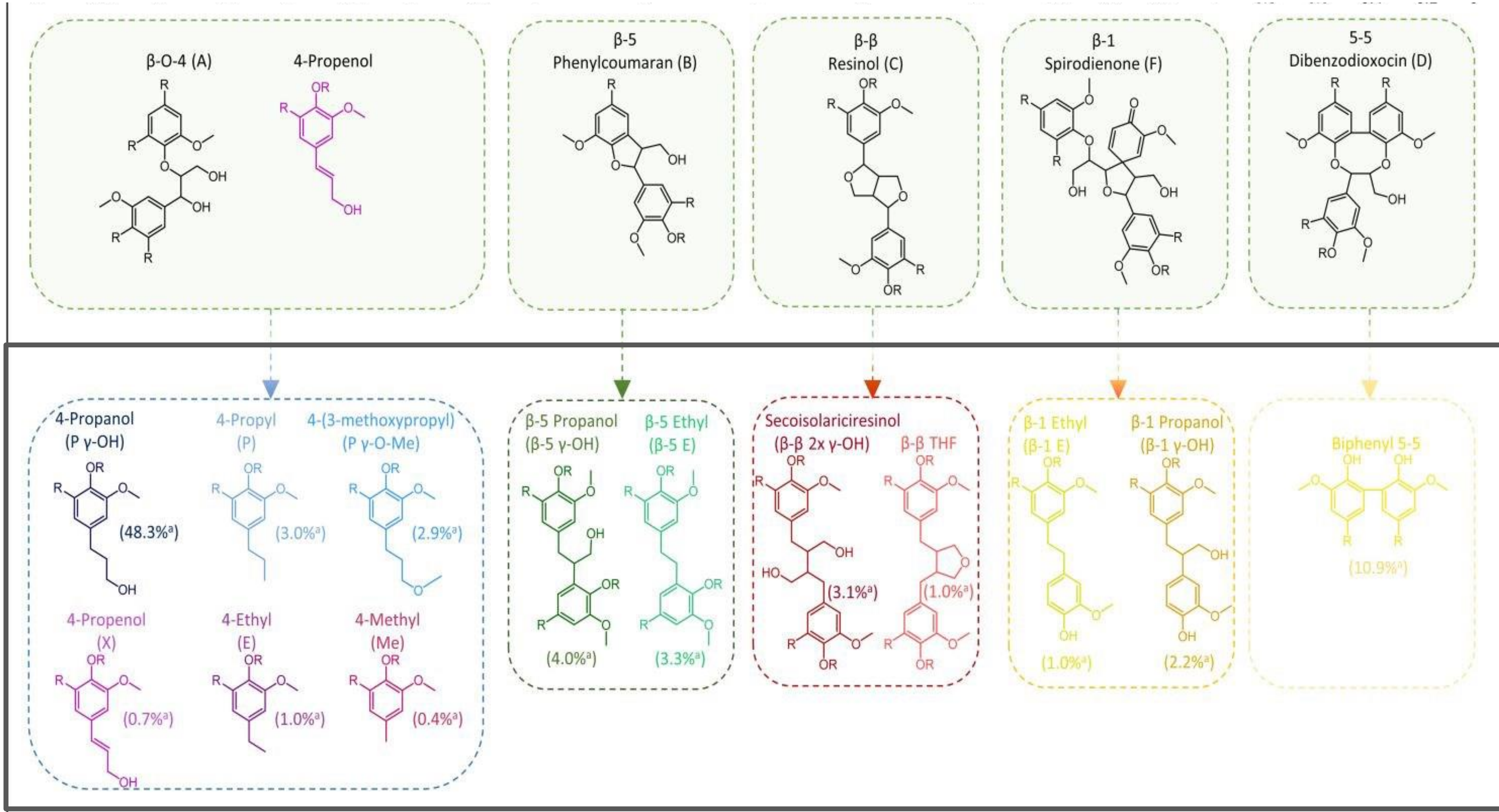


Van Aelst et al. *Chemical Science* 2020

LIGNIN OIL OLIGOMER FUNCTIONALITY AND MOLECULAR STRUCTURES

KATHOLIEKE UNIVERSITEIT LEUVEN

> 80% interlinkages and end groups were recently identified



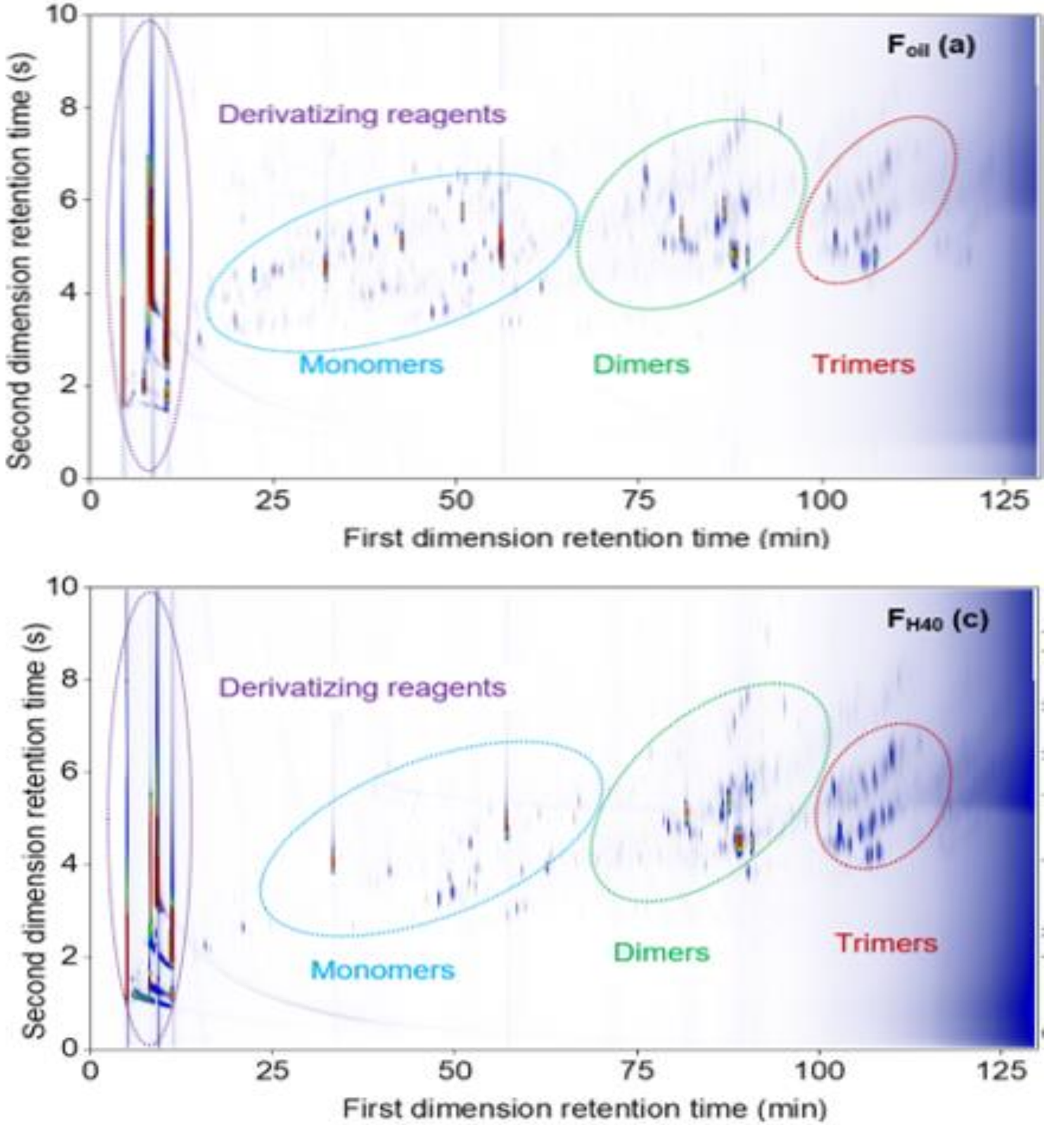
Van Aelst K *et al*. *Chemical Science* 2020

LIGNIN OIL OLIGOMERS MOLECULES IDENTIFICATION: HT-GCXGC-MS/FID

K. Van Geem University Ghent

Lignin Oil

Oligomer fraction

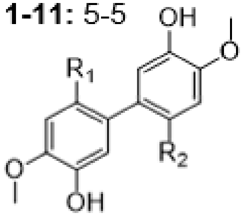


Thi D D and Van Aelst K et al. *Green Chemistry*, 2022.

ILLUSTRATION OF IDENTIFIED DIMERS

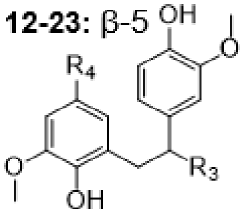
KATHOLIEKE UNIVERSITEIT LEUVEN

1-11: 5-5



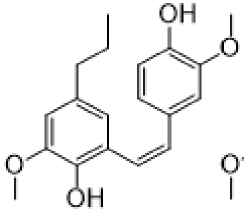
1: $R_1 = CH_3$; $R_2 = (CH_2)_2CH_2OH$
2: $R_1 = R_2 = (CH_2)_2CH_2OH$
3: $R_1 = (CH_2)_2CH_3$; $R_2 = (CH_2)_2CH_2OCH_3$
4: $R_1 = CH_2CH_3$; $R_2 = (CH_2)_2CH_2OH$
5: $R_1 = (CH_2)_2CH_2OCH_3$; $R_2 = (CH_2)_2CH_2OCH_3$
6: $R_1 = (CH_2)_2CH_3$; $R_2 = CH_2CH_3$
7: $R_1 = R_2 = (CH_2)_2CH_3$
8: $R_1 = (CH_2)_2CH_3$; $R_2 = (CH_2)_2CH_2OH$
9: $R_1 = R_2 = CH_2CH_3$
10: $R_1 = CH_2CH_3$; $R_2 = CH_3$
11: $R_1 = (CH_2)_2CH_2OH$; $R_2 = (CH)_2CH_2OH$

12-23: β-5

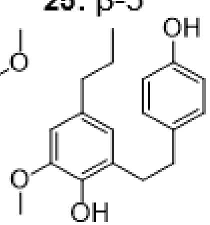


12: $R_3 = CH_2OH$; $R_4 = (CH_2)_2CH_3$
13: $R_3 = CH_2OH$; $R_4 = (CH_2)_2CH_2OH$
14: $R_3 = CH_2OH$; $R_4 = CH_3$
15: $R_3 = R_4 = H$
16: $R_3 = CH_3$; $R_4 = (CH_2)_2CH_2OH$
17: $R_3 = CH_2OH$; $R_4 = (CH_2)_2CH_2OCH_3$
18: $R_3 = H$; $R_4 = (CH_2)_2CH_3$
19: $R_3 = CH_3OH$; $R_4 = CH_2CH_3$
20: $R_3 = H$; $R_4 = (CH_2)_2CH_2OH$
21: $R_3 = CH_3$; $R_4 = (CH)_2CH_2OCH_3$
22 $R_3 = H$; $R_4 = (CH)_2CH_2OCH_3$
23: $R_3 = CH_3$; $R_4 = (CH_2)_2CH_3$

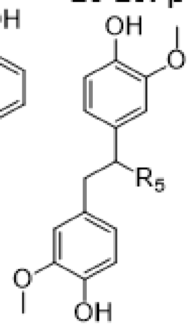
24: β-5



25: β-5

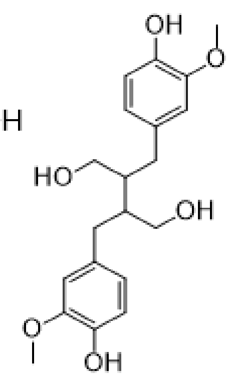


26-28: β-1

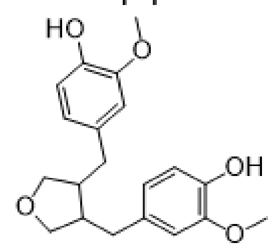


26: $R_5 = CH_3$
27: $R_5 = H$
28: $R_5 = CH_2OH$

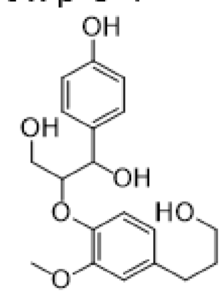
29: β-β



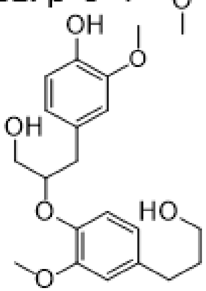
30: β-β



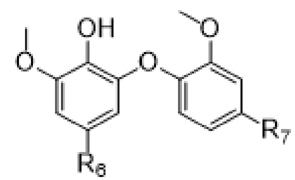
31: β-O-4



32: β-O-4



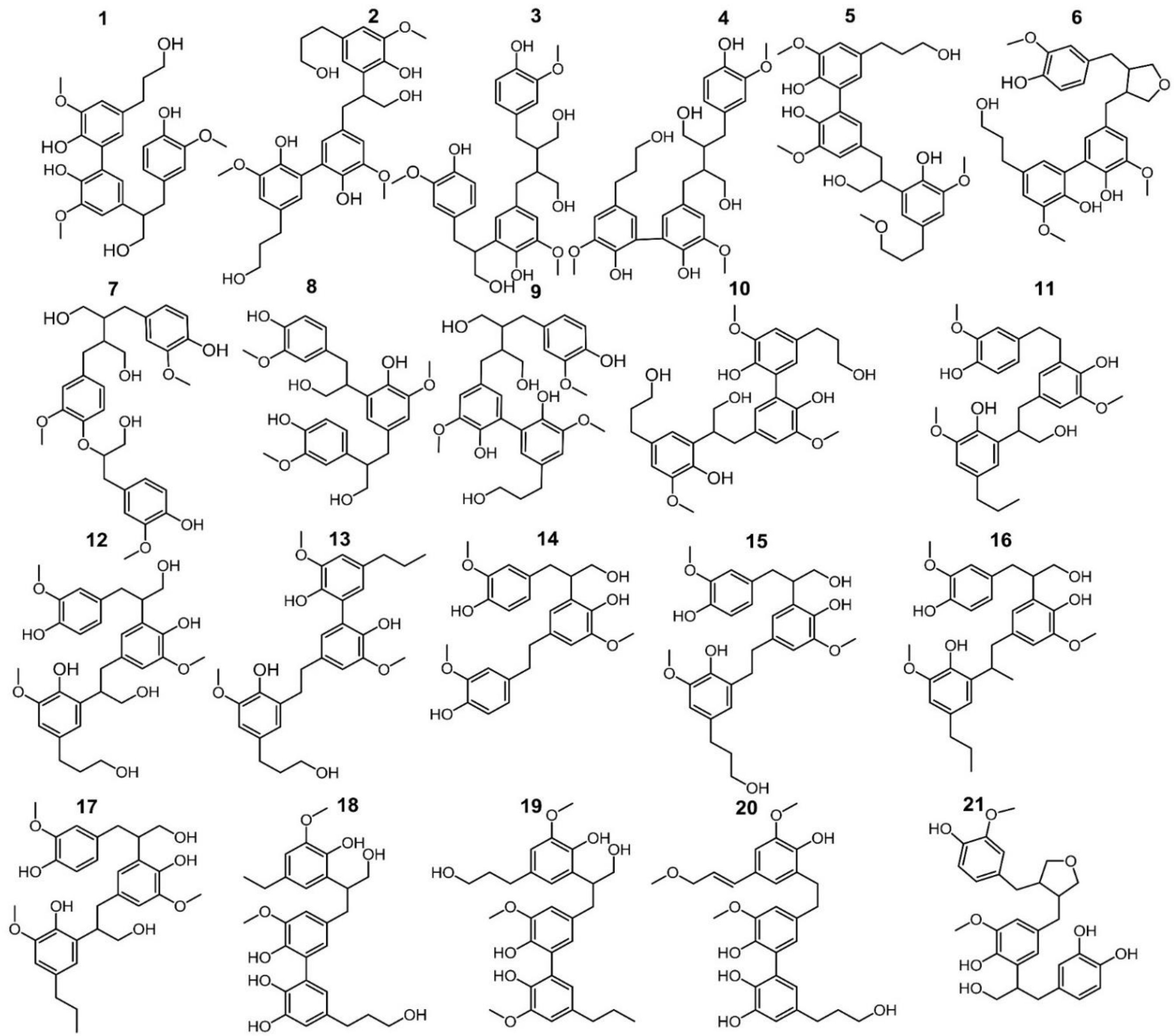
33-36: 4-O-5



33 & 34: $R_6 = R_7 = (CH_2)_2CH_2OH$
35: $R_6 = (CH_2)_2CH_2OCH_3$; $R_7 = (CH)_2CH_2OCH_3$
36: $R_6 = (CH_2)_2CH_3$; $R_7 = (CH)_2CH_2OH$

Thi D D and Van Aelst K et al. *Green Chemistry*, 2022.

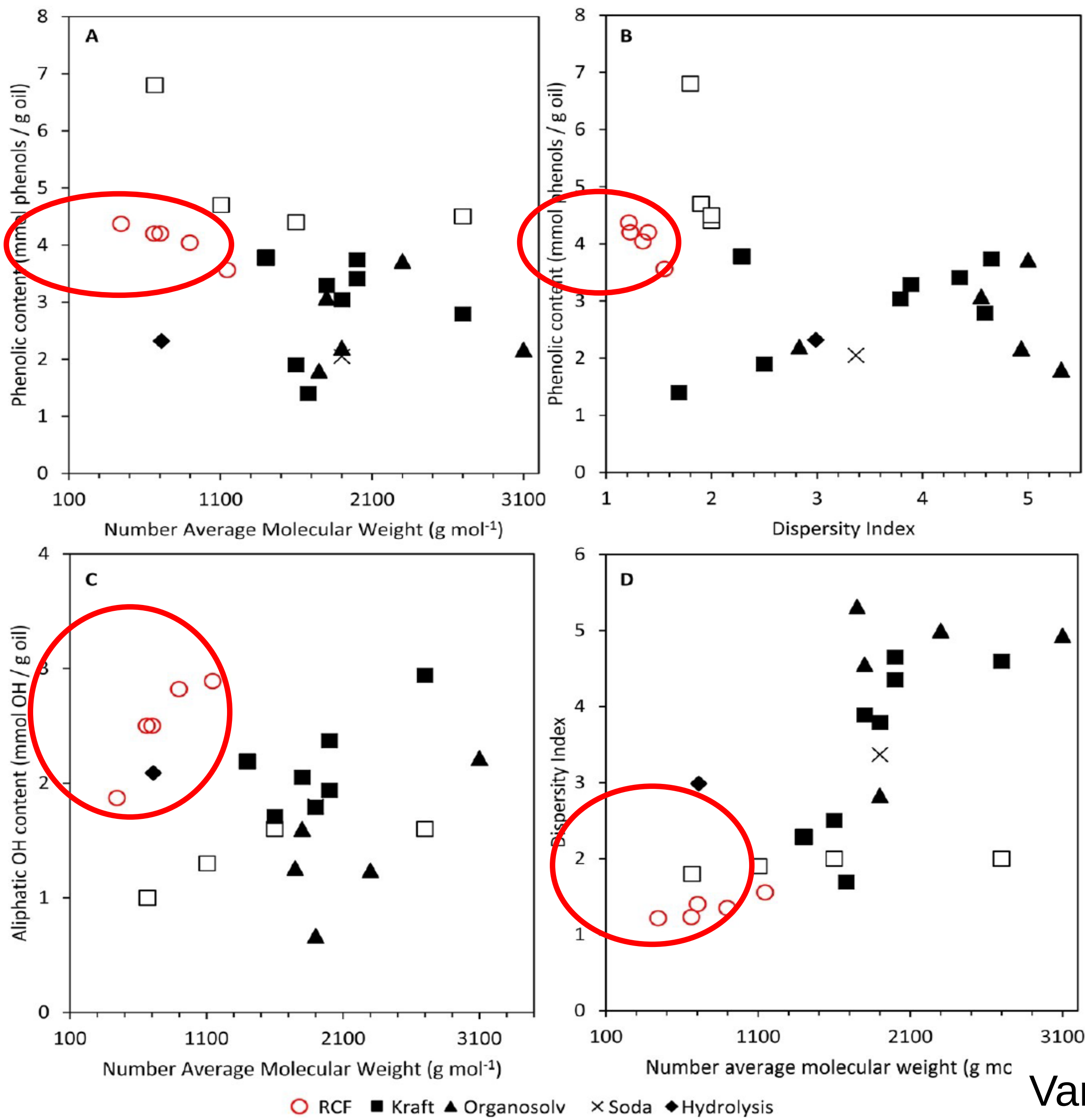
ILLUSTRATION OF IDENTIFIED TRIMERS



Thi D D and Van Aelst K et al. *Green Chemistry*, 2022.

RCF LIGNIN OILS COMPOSITION POSITIONS UNIQUE FEATURES FOR VARIOUS APPLICATIONS

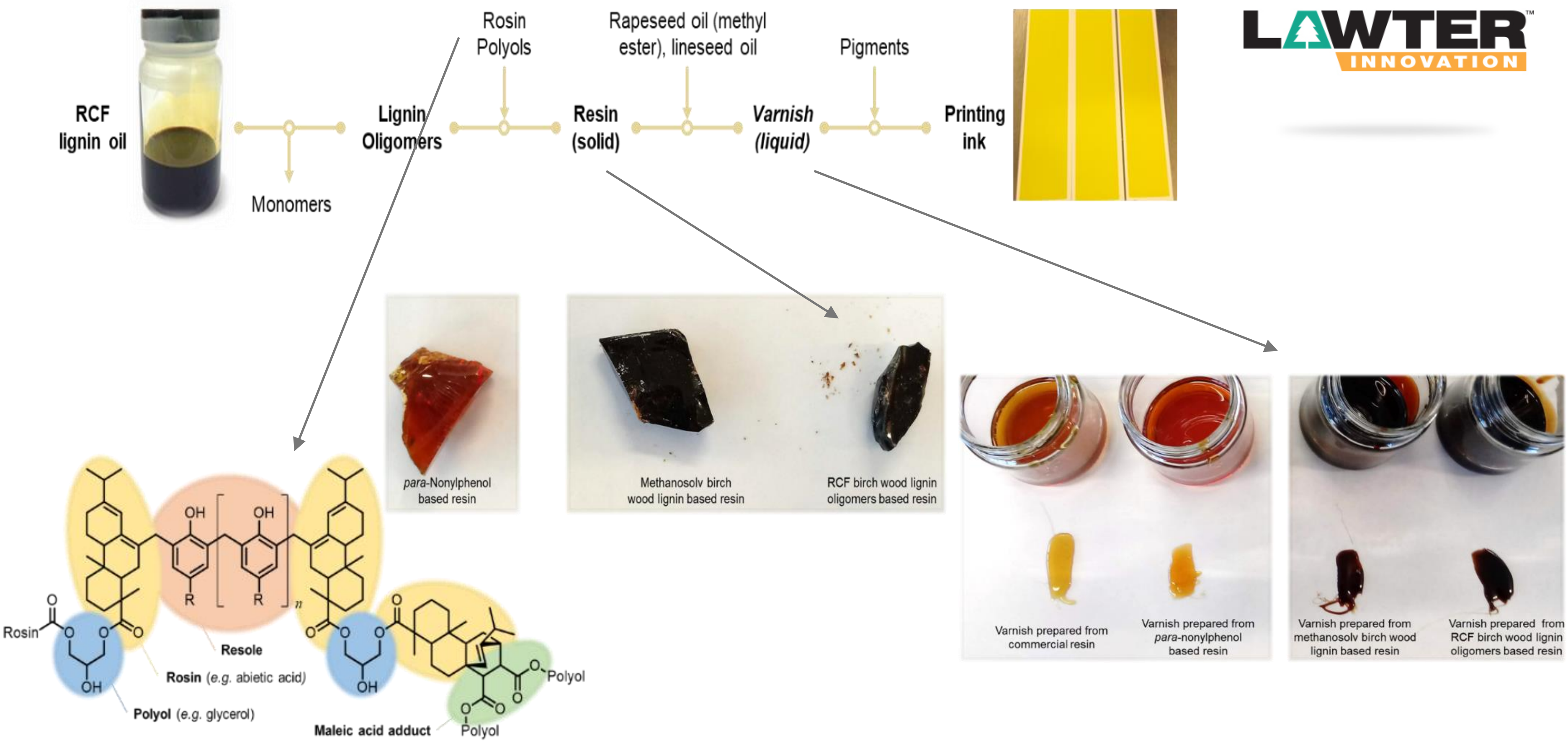
KATHOLIEKE UNIVERSITEIT LEUVEN



Low dispersity D
Low molecular weight MW
High functionality
(aliphatic OH)

Van Aelst K. *Chem Commun* 202

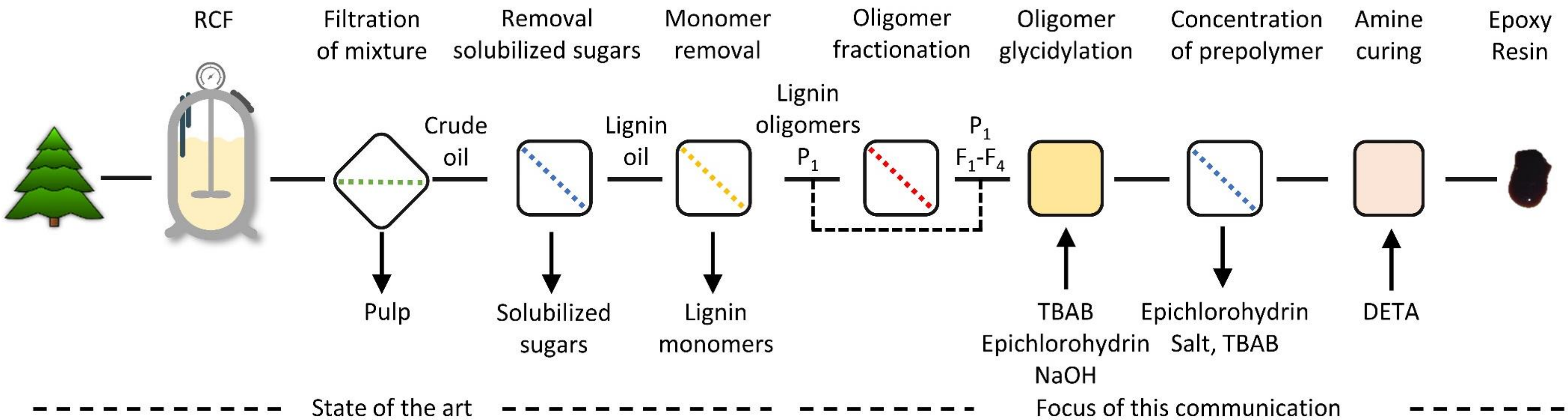
EXAMPLE 1: LIGNIN OIL OLIGOMERS TO RESINS, VARNISHES AND PRINTING INK

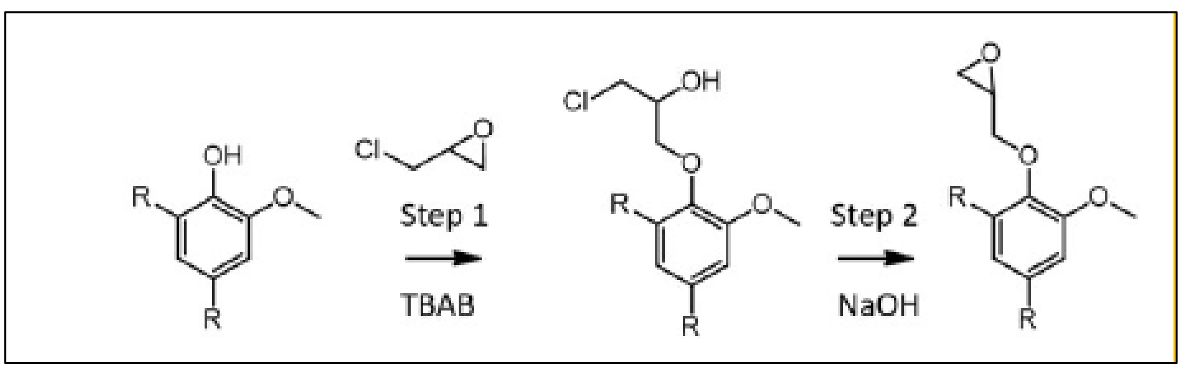


Replacing endocrinic nonylphenol oligomers

Liao et al. *Science* 2020

EXAMPLE 2: LIGNIN OIL OLIGOMERS TO EPOXY RESINS, PURS, …

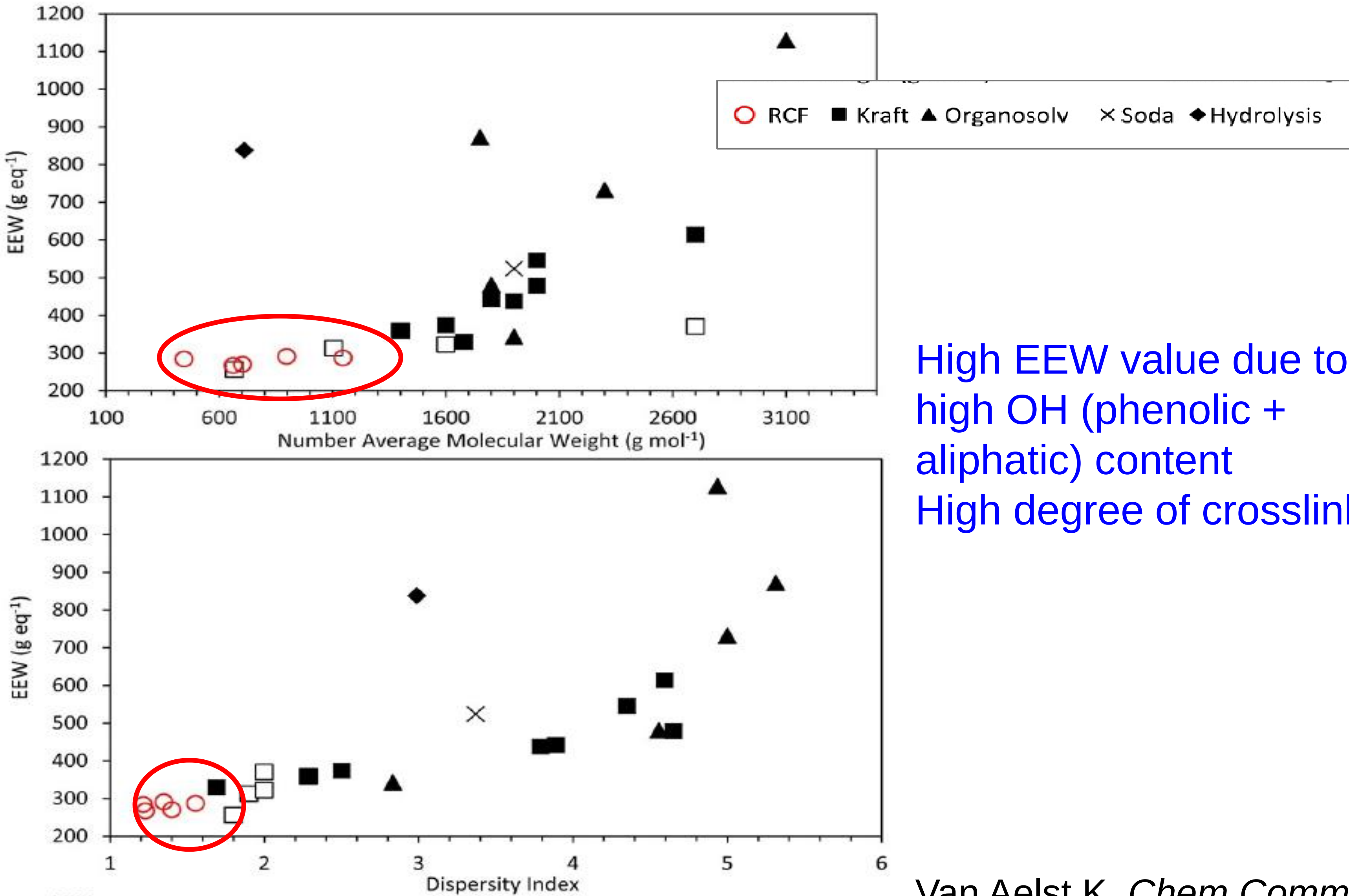




? Functionalisation of phenols or aliphatic OHs

Van Aelst K. *Chem Commun* 202

EXAMPLE 2: LIGNIN OIL OLIGOMERS TO EPOXY RESINS – HIGH EPOXIDE CONTENT



High EEW value due to high OH (phenolic + aliphatic) content

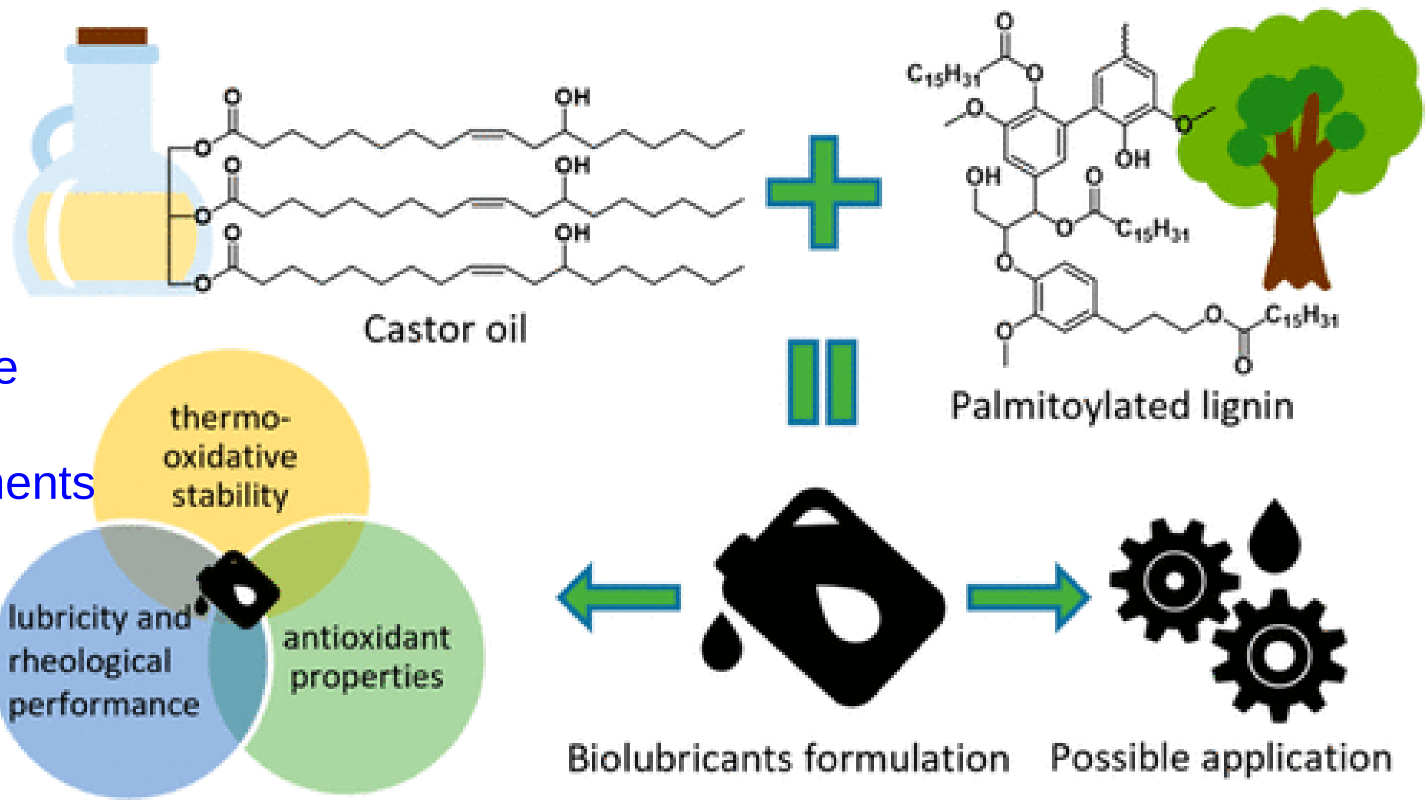
High degree of crosslinking

Van Aelst K. *Chem Commun* 20

KATHOLIEKE UNIVERSITEIT LEUVEN

EXAMPLE 3: LIGNIN OIL OLIGOMERS TO BIOLUBRICANTS

With K. Bernaerts University Maastricht



Stable lignin fragments

High fatty acid content

High free phenolic content

Jedrzejczyk et al. *ACS Sust Chem & Eng*, 2021

TAKE HOME MESSAGES

Lignin is still exciting research domain

Lignin is a biomass feedstock. Its usage can add to the sustainability goals

Lignin has large potential due to

- Better molecular understanding
- Development of new biorefineries producing novel lignins
- That is capable of tailoring lignin properties

Ready for upscaling adventures

Integration in chemical industry as at the doorstep

Validate process-technical, economical and sustainability measures

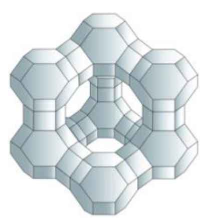
CURRENT RESEARCH GROUP

KATHOLIEKE UNIVERSITEIT LEUVEN

Post-docs:
Dr. Sander Van den Bosch (PDM)
Fatima Rammal
Ivan Scodeller
Guillaume Pomolaza
José Luis Santos Muñoz
Xuejiao Wu
Thijs Van Geel (VLAIO)
Dieter Plessers (FWO)
Ghinwa Fayad
Korneel Van Aelst
Franscesco Brandi
Deepak Raikwar

Project and Lab Manager:
Dr. Ekaterina Makshina (INDU)

Technicians:
Walter Vermandel (INDU)
Ben Wambacq
Stef Use
Werner Wouters
Johan Maes



Valorisation and Innovation Managers
Dr. ir. Bert Lagrain (IOF)
Dr. ir. Joost Van Aelst (VLAIO)

PhDs:
Bert Boekaerts (VLAIO)
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