

Transformation of lignocellulose into aromatic building blocks

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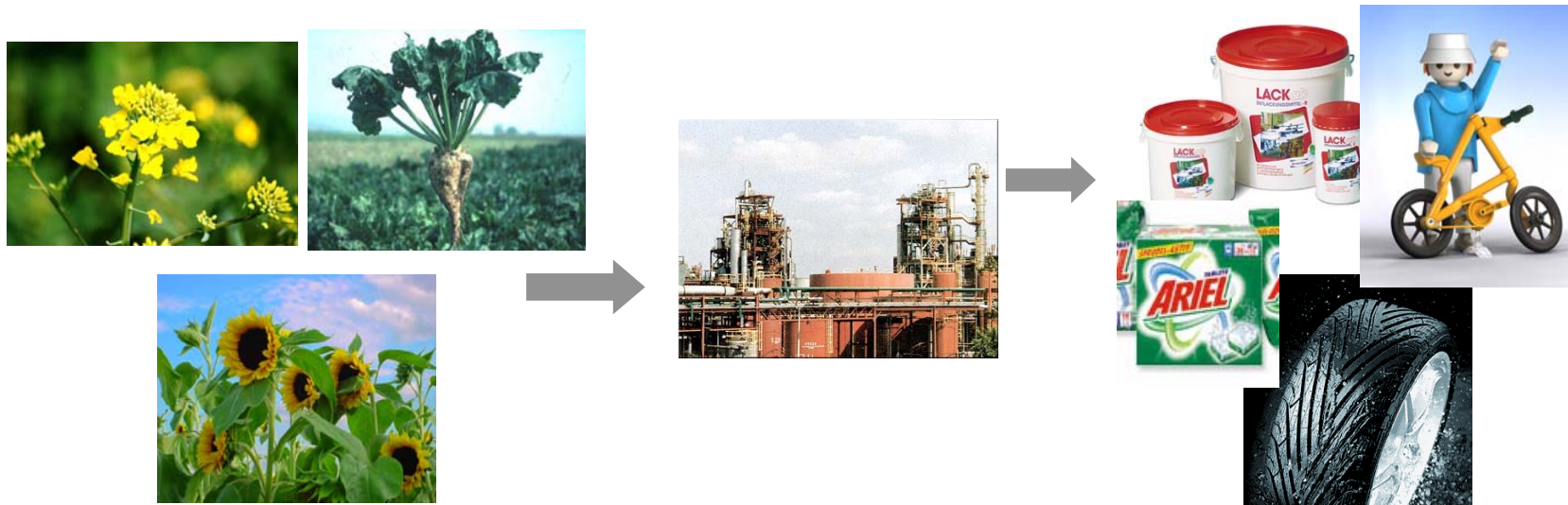
Introduction

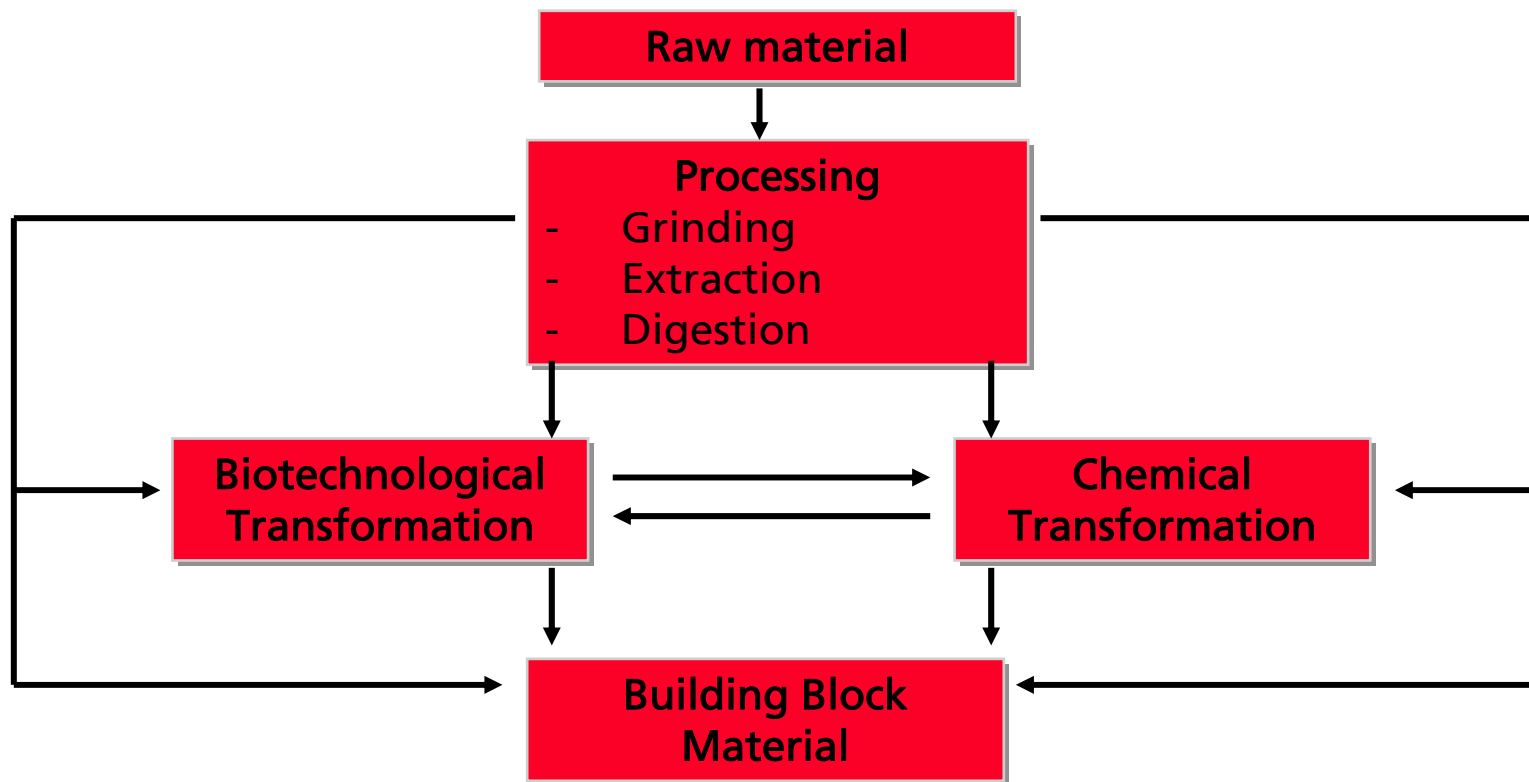
- Potential of Lignocellulose-Feedstock's (LCF) -



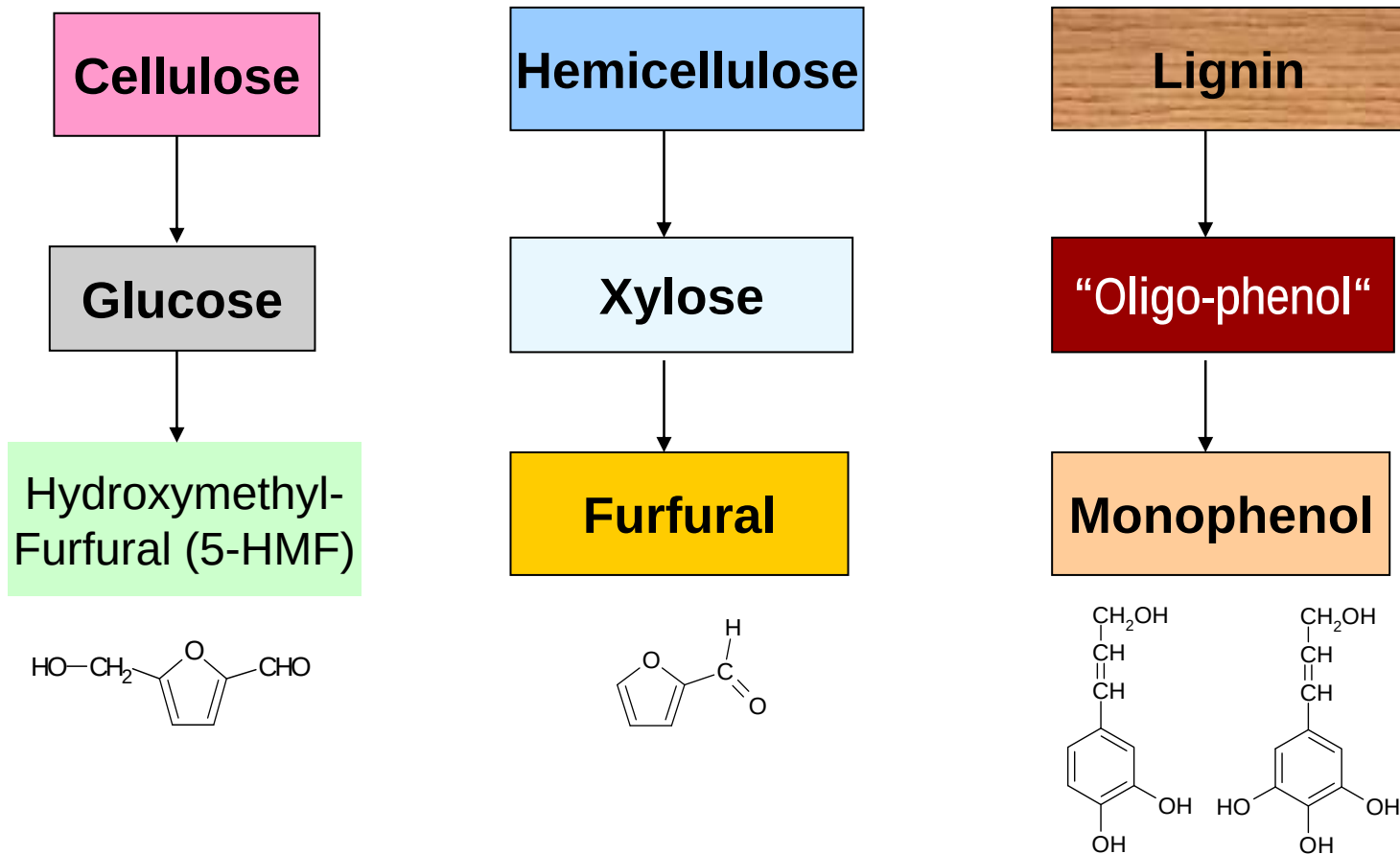
Vision

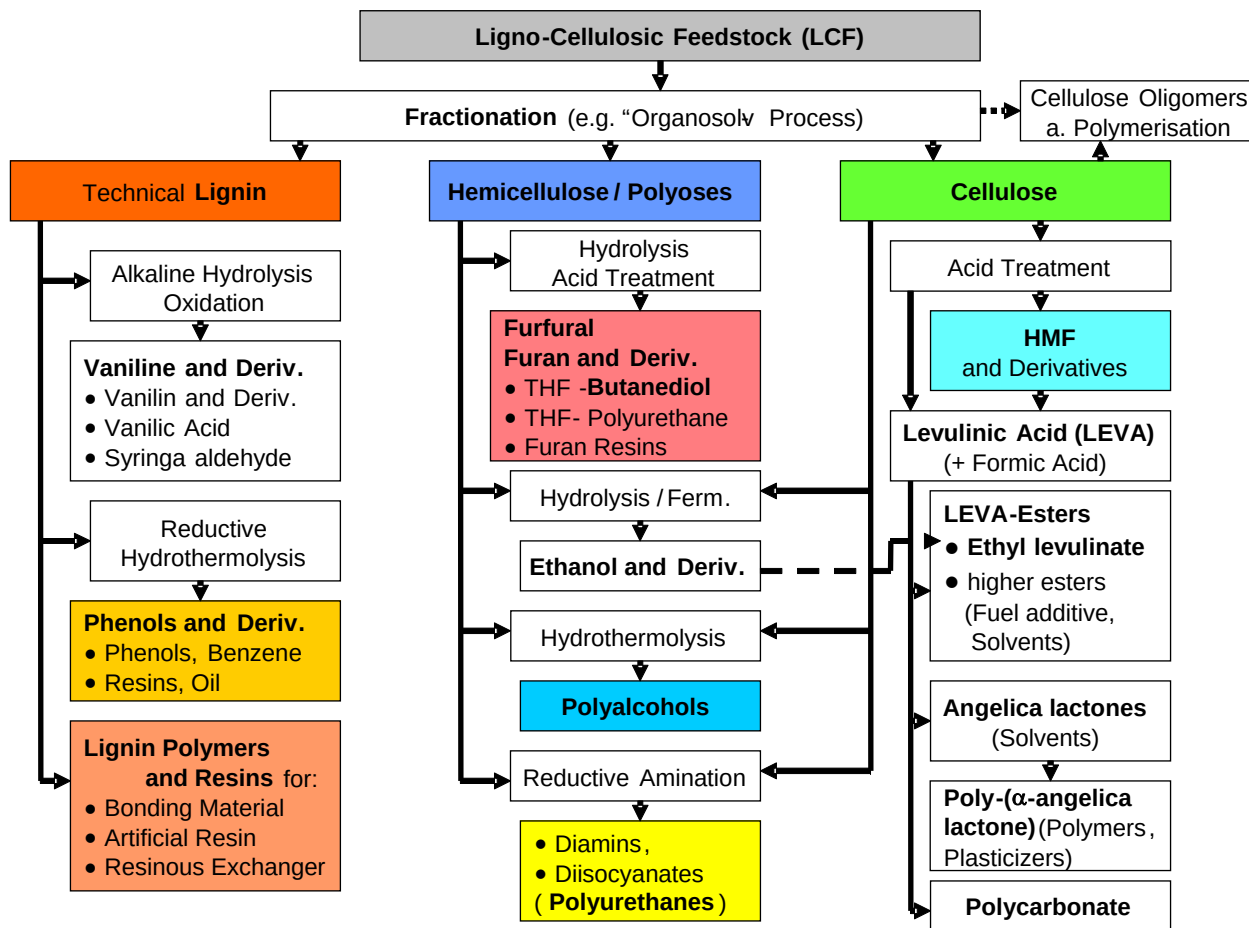
Exploration of new synthesis strategies and development of new production sites for the partial substitution of fossil to renewable resources within the middle of the 21 century.





From feedstock to product





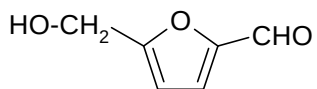
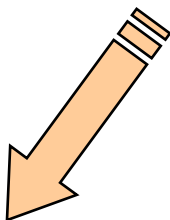
Source: DOW

Sugar based aromatics



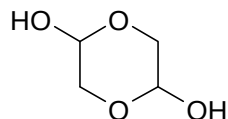
Glucose, Fructose

Hydrothermolysis



5-HMF

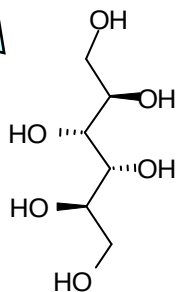
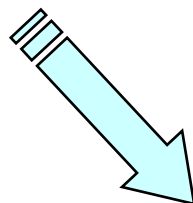
5-Hydroxymethylfurfural



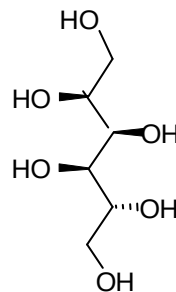
DHD

2,5-Dihydroxydioxan

reductive
Hydrothermolysis

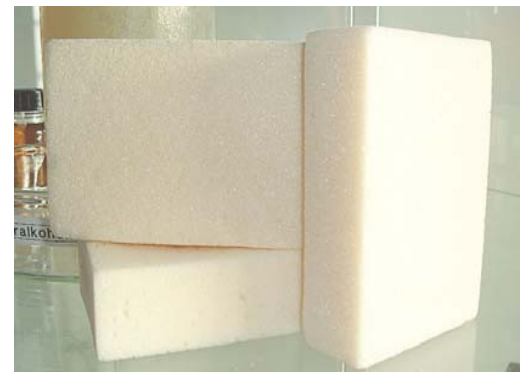


Mannit

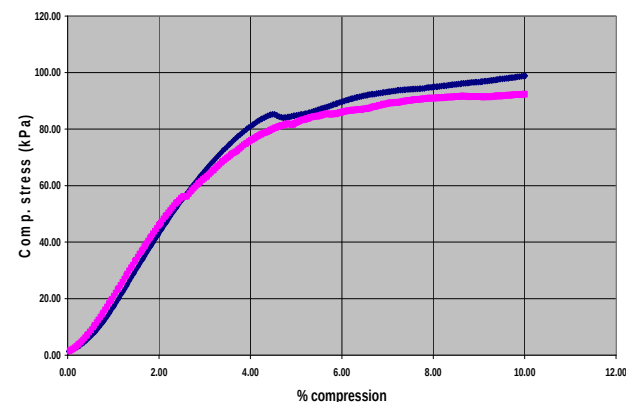


Sorbit

Polyalcohols

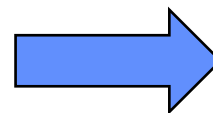
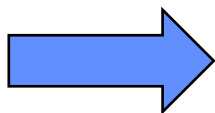


Compressive strength _ rise for reference #1 and formulation #3



• Reference #1 • Formulation #3

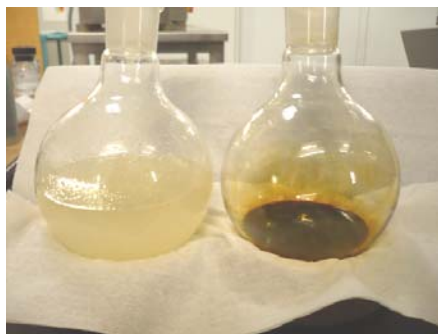
Purification of 5-Hydroxymethylfurfural via distillation



5-HMF
(raw)

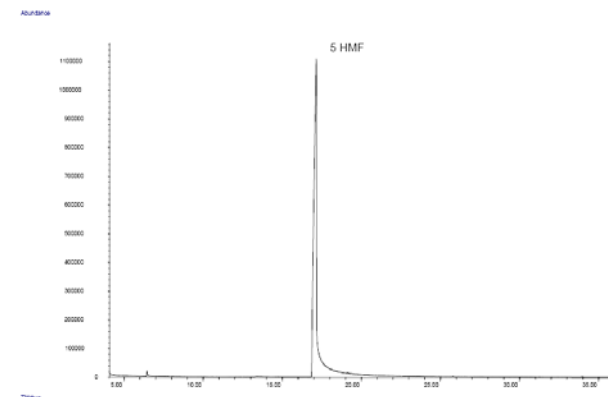
5-HMF
(purified)

residue



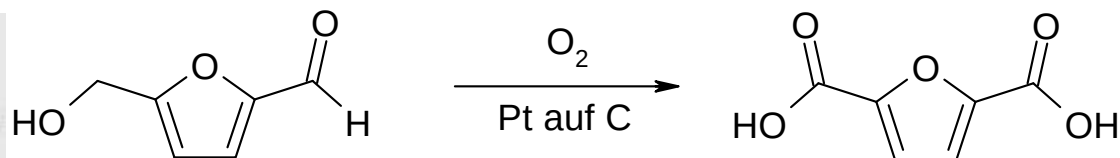
HMF
(raw material)

residue



DBU-Vorhaben
„Synthesebausteine aus
nachwachsenden
Rohstoffen,“
Aktenzeichen: 24621

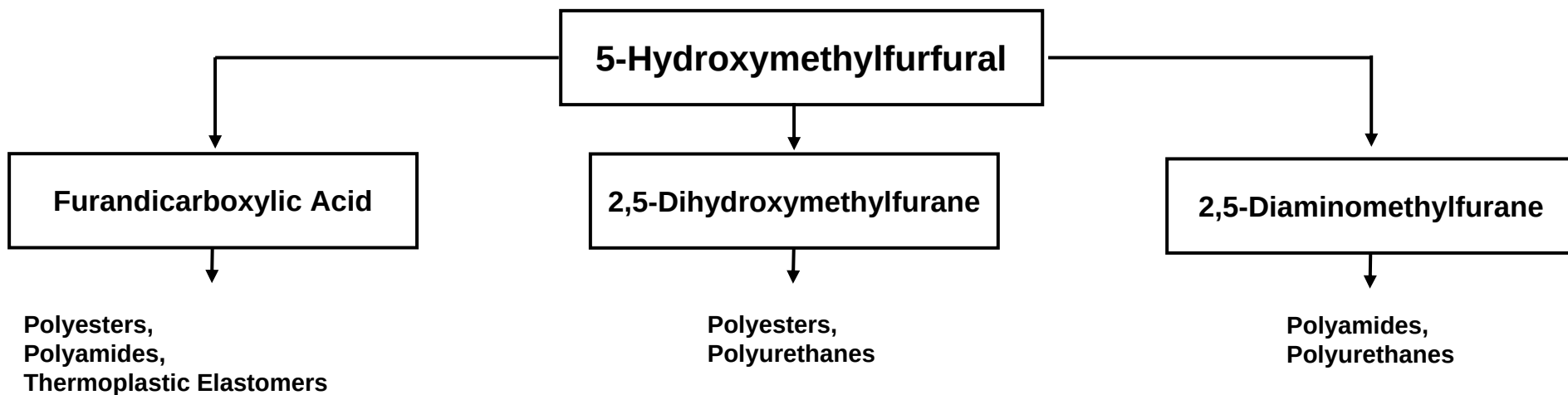
ICT Synthesis of 2,5-Furandicarboxylic acid (FDCA)



Oxidation of 5-Hydroxymethylfurfural in water

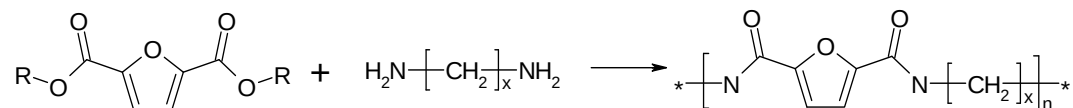
Batch size max. 2,5 kg 5-HMF

Yield FDCA > 98 %

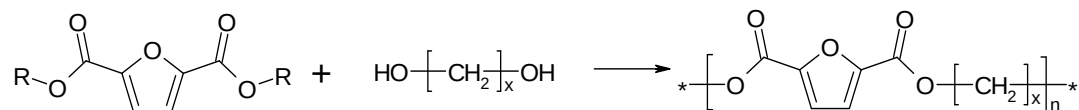


Biopolymers based on Furanedicarboxylic acid

Polyamides



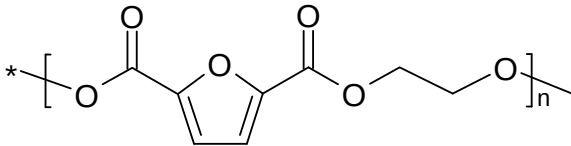
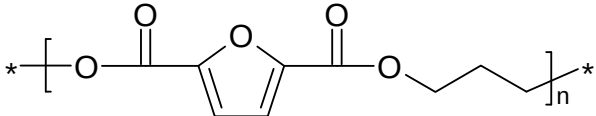
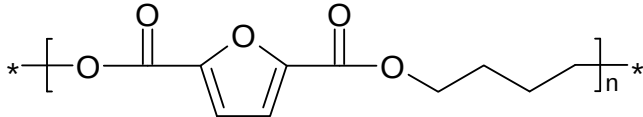
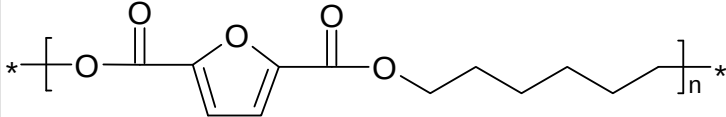
Polyesters



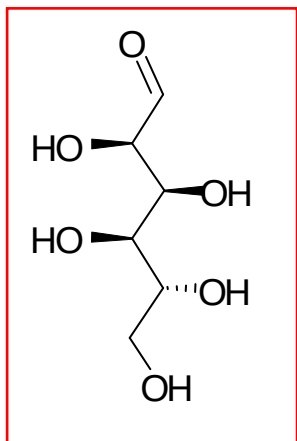
Thermal properties polyamides of glucose based polyamides

$* \left[\text{NH} - \text{C}(=\text{O}) - \text{C}_5\text{H}_3\text{O} - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_4 \right]_n *$	$T_m = 250\text{ }^{\circ}\text{C}$
$* \left[\text{NH} - \text{C}(=\text{O}) - \text{C}_5\text{H}_3\text{O} - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_6 \right]_n *$	$T_m = 175\text{ }^{\circ}\text{C}$ Fibres
$* \left[\text{NH} - \text{C}(=\text{O}) - \text{C}_5\text{H}_3\text{O} - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_8 \right]_n *$	$T_m = 125\text{ }^{\circ}\text{C}$

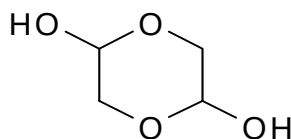
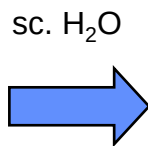
Thermal properties of glucose based polyesters

 <chem>*[O]C(=O)c1cc(OC(=O)OCCO)cc1</chem>	$T_m = 205-210\text{ }^{\circ}\text{C}$ Fibres from melt
 <chem>*[O]C(=O)c1cc(OC(=O)OCC)cc1</chem>	$T_m = 120\text{ }^{\circ}\text{C}$
 <chem>*[O]C(=O)c1cc(OC(=O)OCCC)cc1</chem>	$T_m = 163 - 165\text{ }^{\circ}\text{C}$
 <chem>*[O]C(=O)c1cc(OC(=O)OCCCCC)cc1</chem>	$T_m = 143 - 146\text{ }^{\circ}\text{C}$

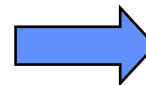
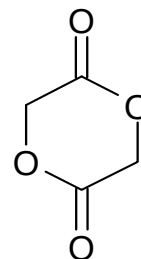
Glucose



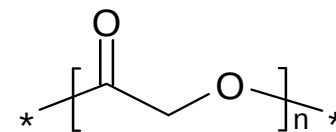
DHD



Oxid.



Polyglykolide



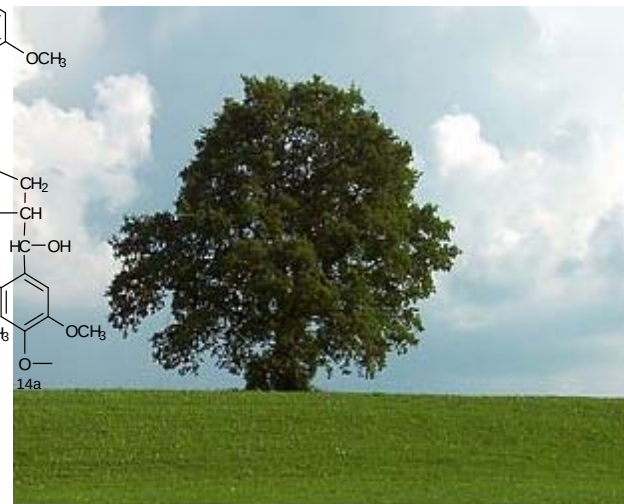
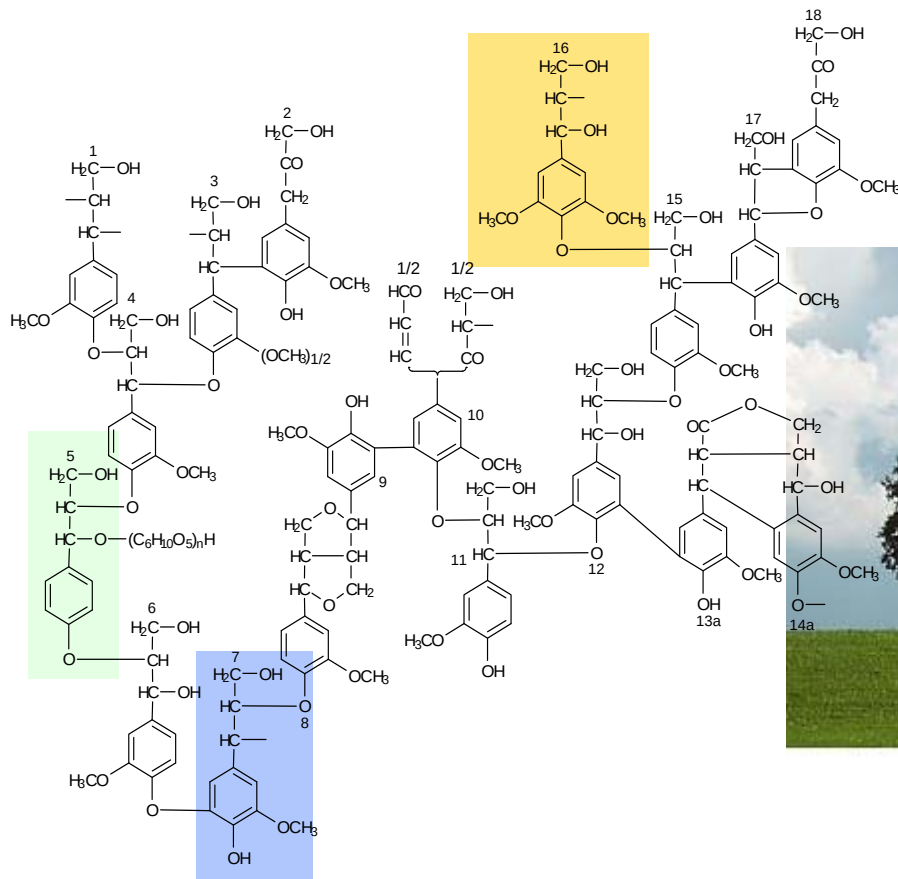
Polyester	T _m (°C)
Poly(3HB)	178
Poly(3HV)	110
Poly(L-lactid)	185
Poly(glycolid)	223-228



Lignin separation and transformation



Lignin



Lignin release from wood

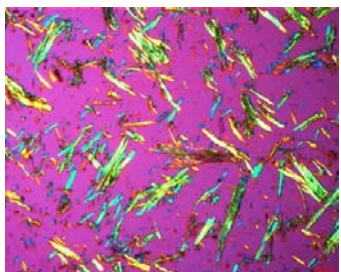
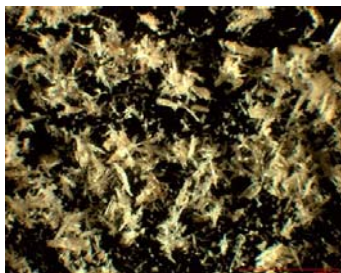
- Sulfate or sulfite process
- Acid wood digestion
- Ionic liquids
- Hydrothermal Treatment (Aquasolv-Process)
- **Organosolv-process (Ethanol, ...)**
- Enzymatic cleavage (+ Fraunhofer IGB)



Lignin production:

The ORGANOSOLV-Process

Temperature: 150 – 250°C
Extraction time: 1 hour
Solvents: Ethanol/Water



Beech wood



Autoclave (13 L)

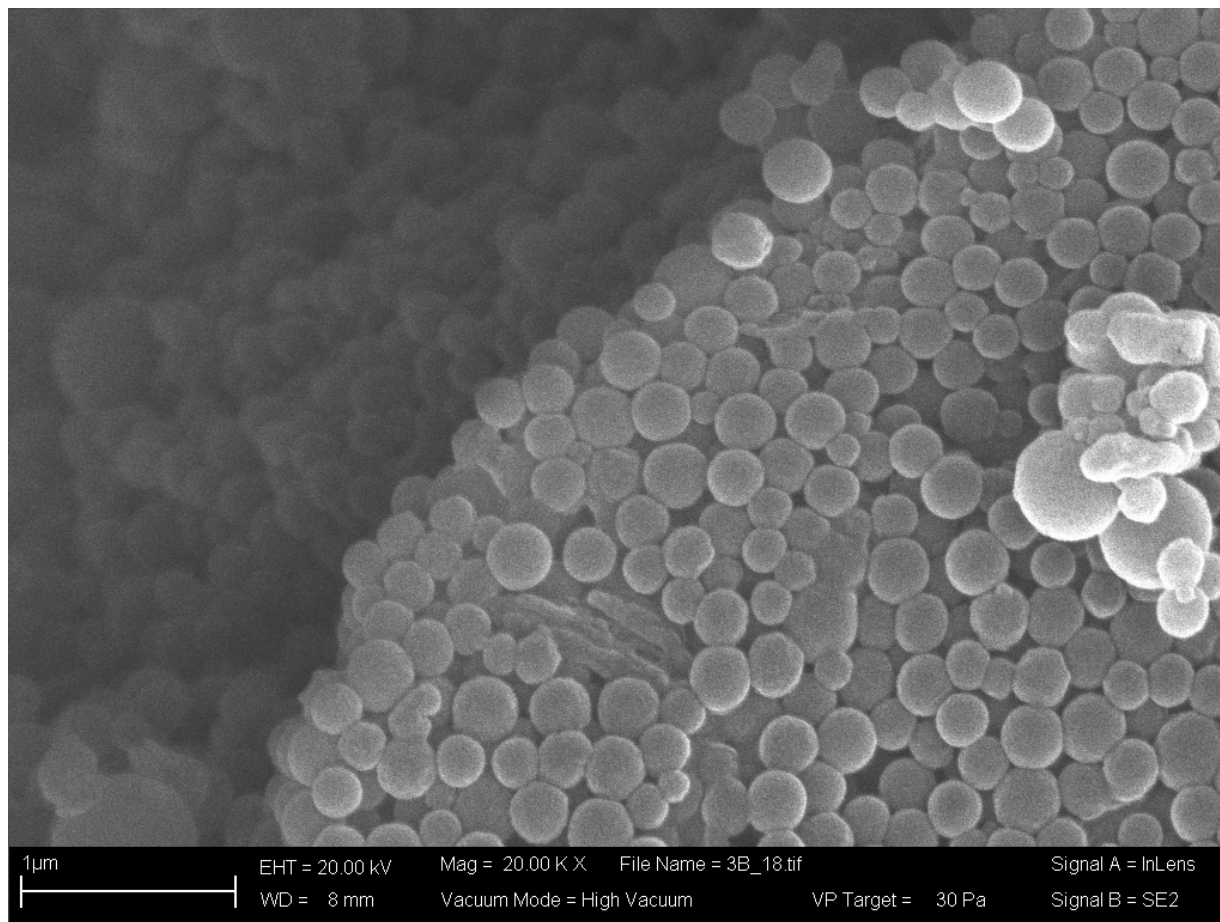


Separation
Solvent Regeneration



Nanosized

Organosolv-Lignin

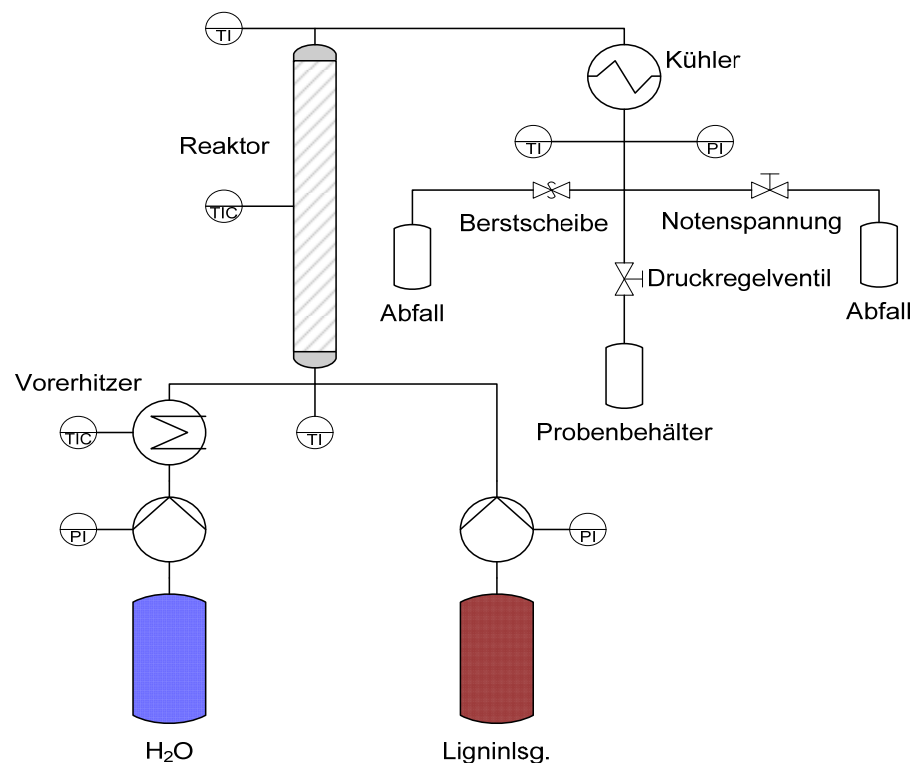


Lignin Transformation: Hydrothermolysis



Process parameter

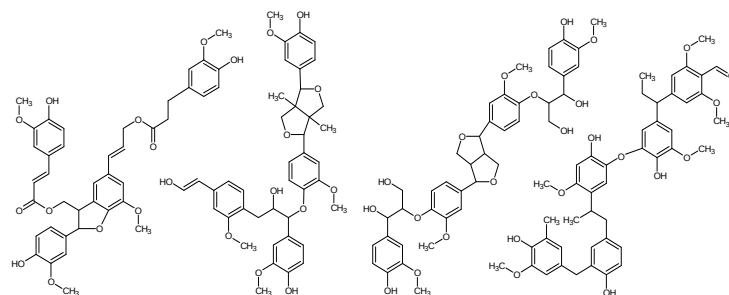
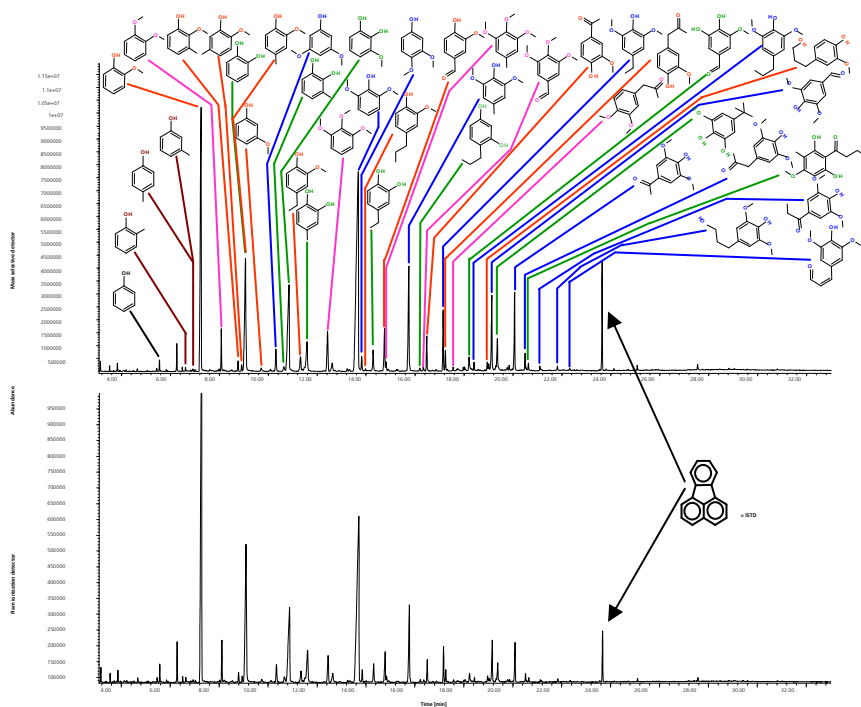
max. temperature:	400°C
max. pressure:	250 bar
max. flow rate:	7,3 L/h
Reactor length:	8- 12 m
Reactor diameter:	9 mm
Residence time:	15 min



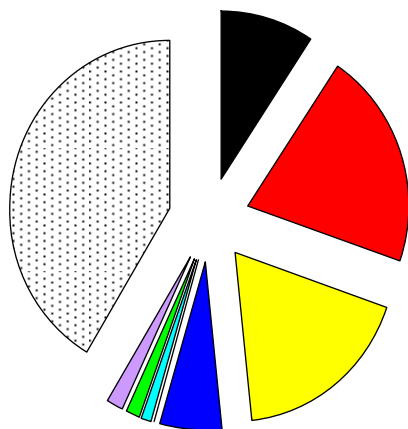
Chemical composition of the products

Fraction of oligomers

Fraction of monomers

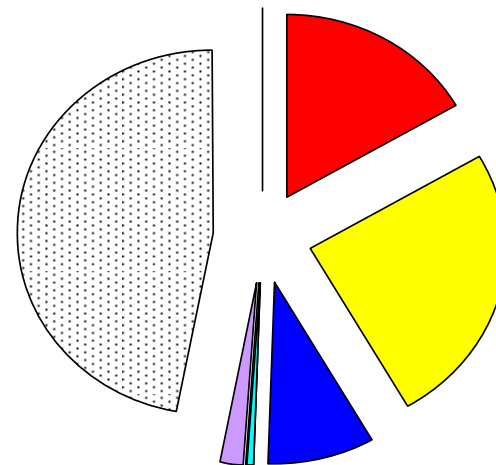


Poplar lignin



- phenol
- guajacol & monomethoxyphenols
- syringol & dimethoxyphenols
- o-hydroxyphenol derivatives
- m-hydroxyphenol derivatives
- benzene; 1,2,3- triol derivatives
- alkylphenols
- di- and trimethoxy-alkylbenzenes; alkylbenzenes
- unknown compounds

Beech wood lignin



Oil composition after hydrothermal treatment

Lignin in composites



Summary and conclusions



- Cellulose, Hemicellulose und Lignin are already used in industrial scales
- Increasing use of Lignocellulose is detectable
- There exist several ways for the production of lignocellulosic products .
- Lignin based products could partially substitute fossil resources. Technical feasibility studies are still running.
- High grade of functionality needs special conversion technologies.



Acknowledgment:

Deutsche Bundesstiftung Umwelt (DBU)

Fachagentur Nachwachsende Rohstoffe (FNR)



Thank you for your attention !