

# Transformation of lignocellulose into aromatic building blocks

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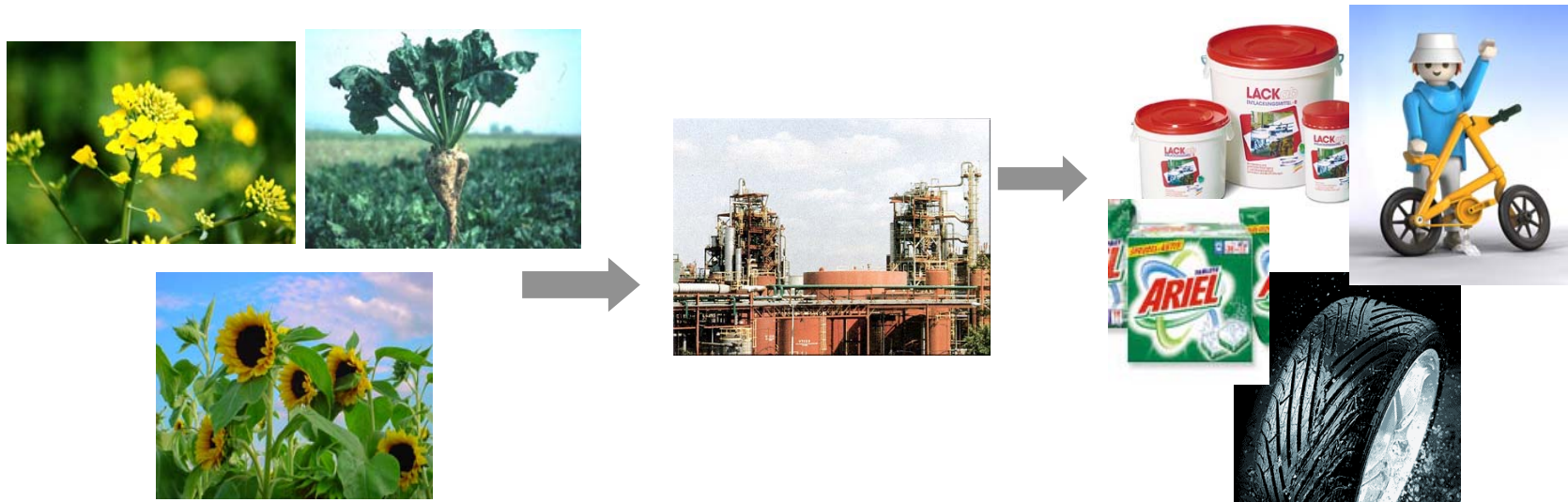
# Introduction

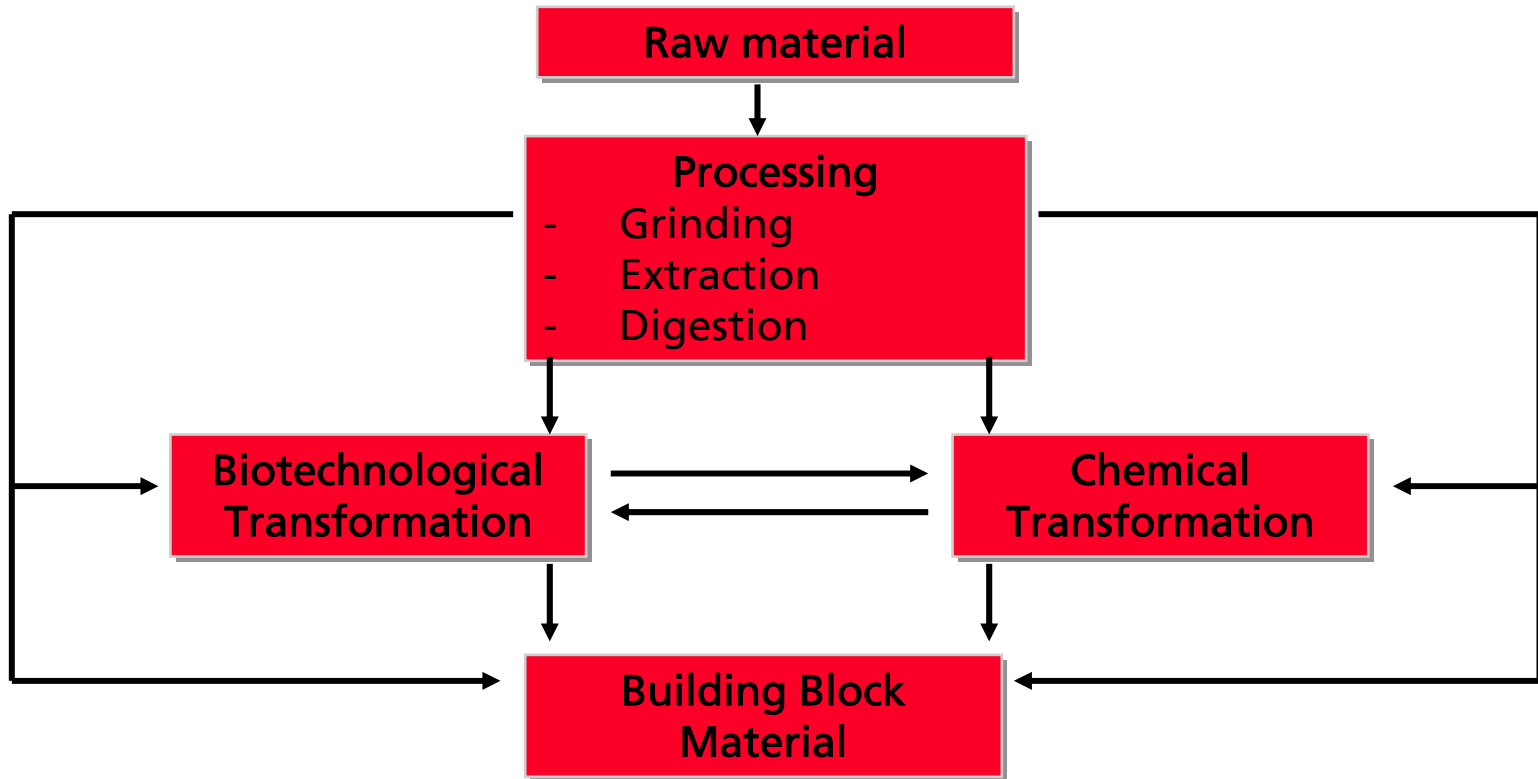
## - Potential of Lignocellulose-Feedstock's (LCF) -



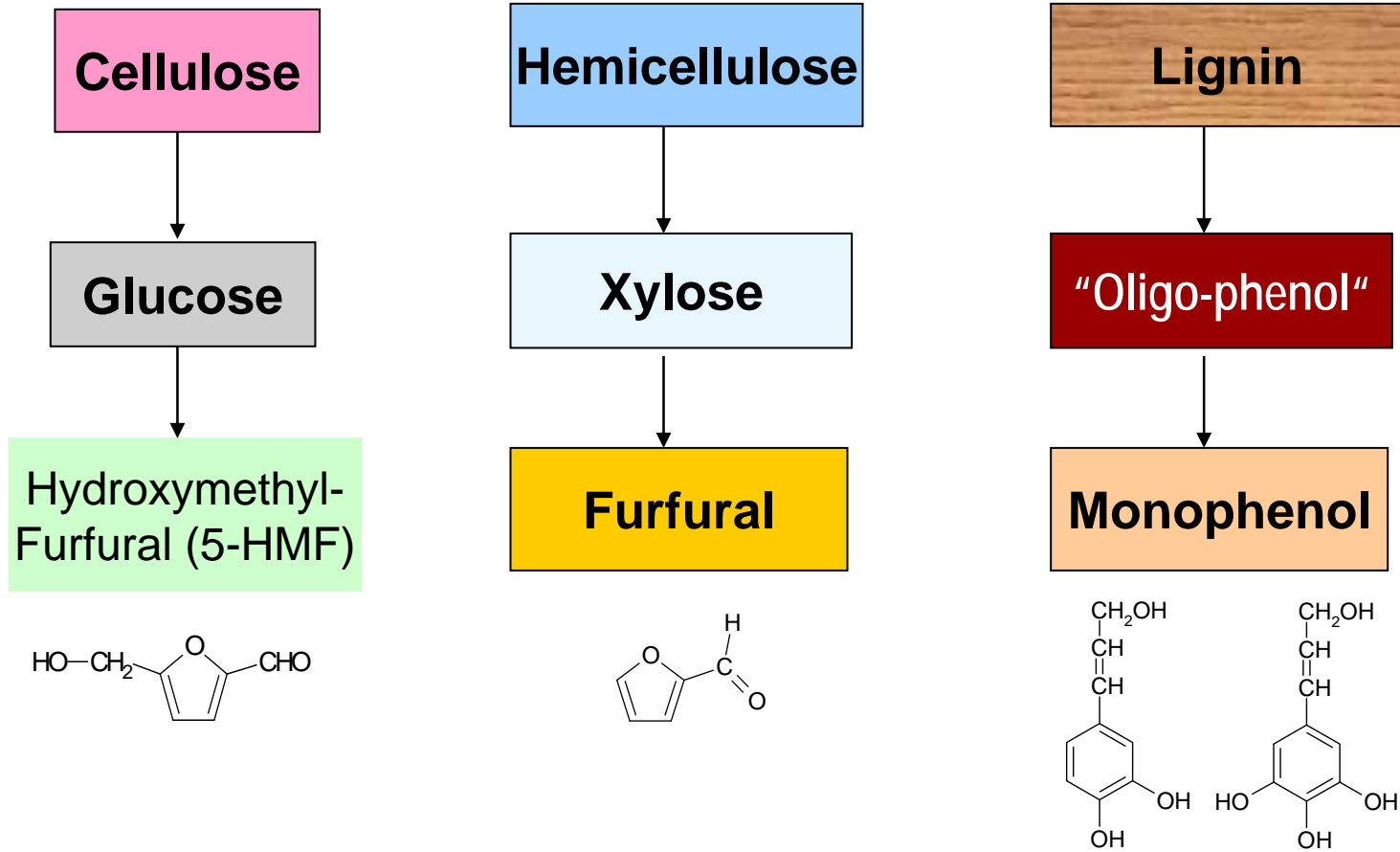
## Vision

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.

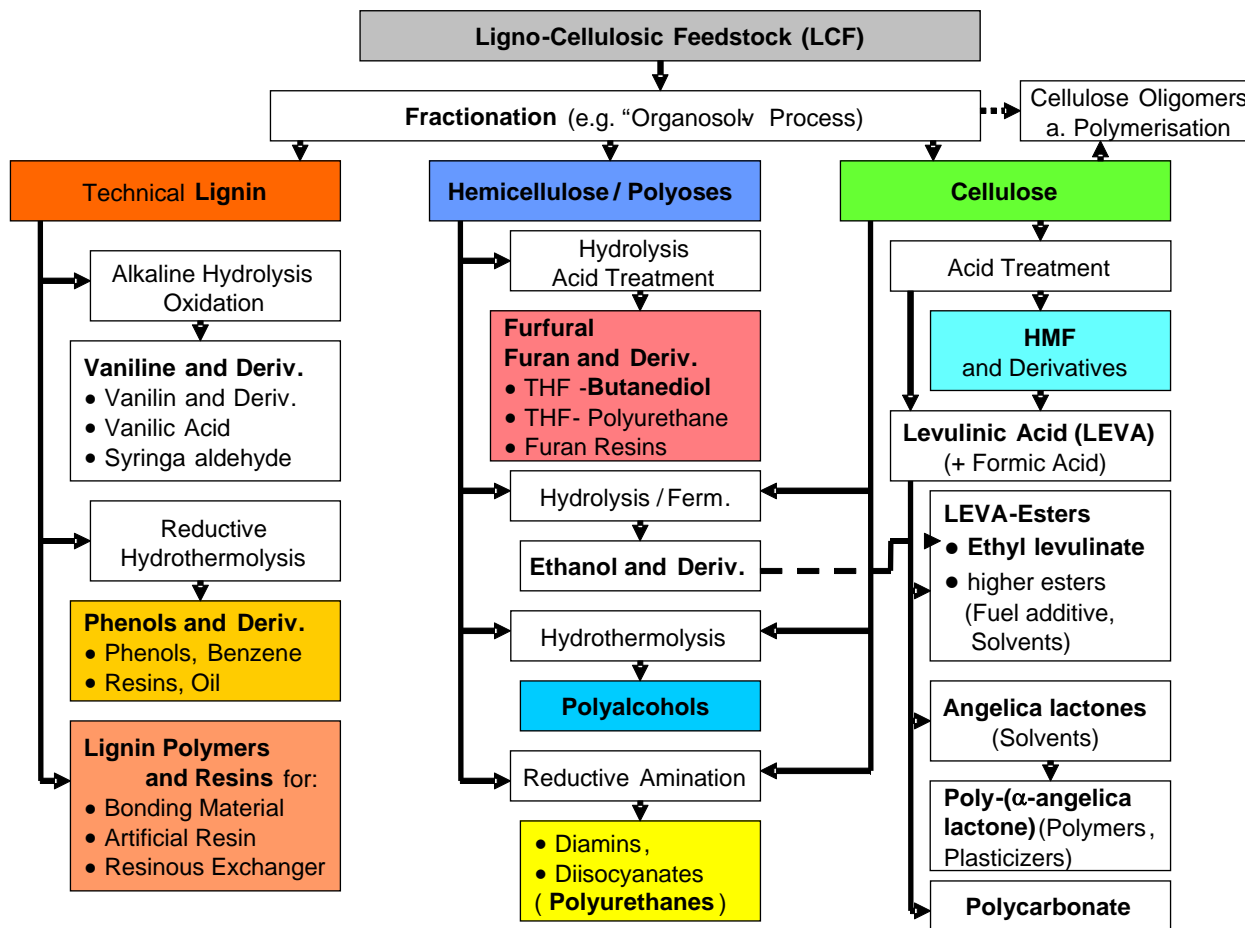




**From feedstock to product**



Transformation of Lignocellulose into aromatic building blocks



Source: DOW

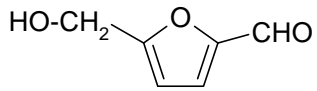
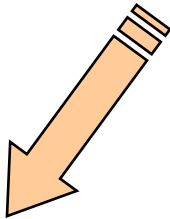
## Sugar based aromatics





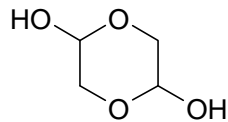
Glucose, Fructose

Hydrothermolysis



5-HMF

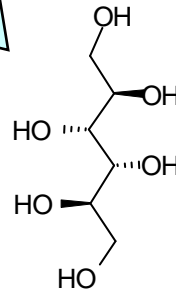
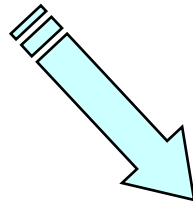
5-Hydroxymethylfurfural



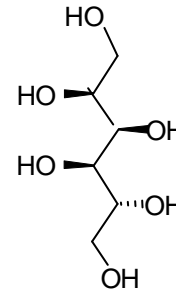
DHD

2,5-Dihydroxydioxan

reductive  
Hydrothermolysis

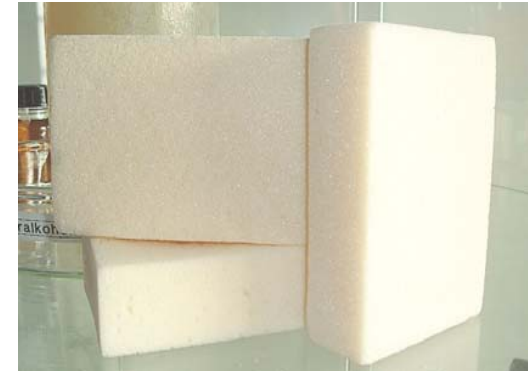


Mannit

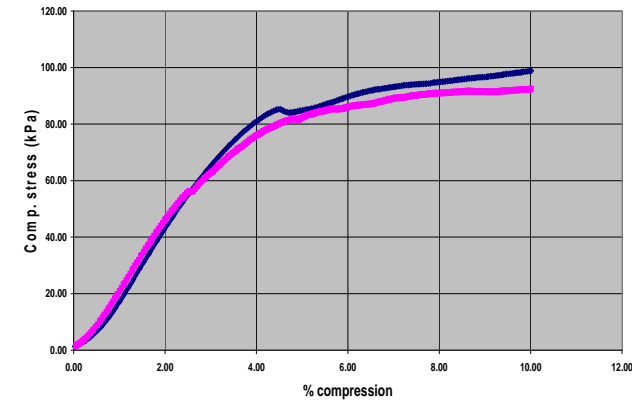


Sorbit

Polyalcohols

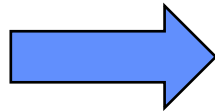


Compressive strength \_|\_ rise for reference #1 and formulation #3



• Reference #1 • Formulation #3

# Purification of 5-Hydroxymethylfurfural via distillation



5-HMF  
(raw)

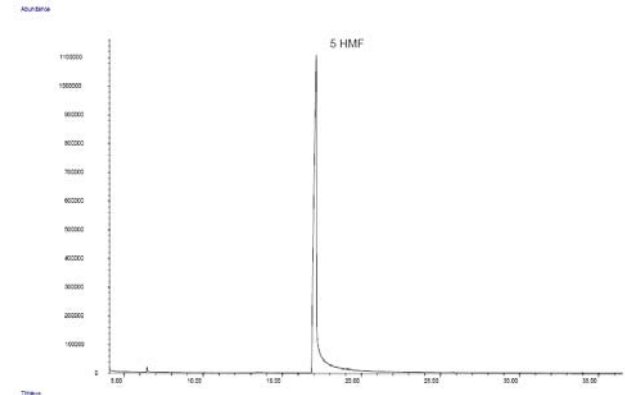
5-HMF  
(purified)

residue



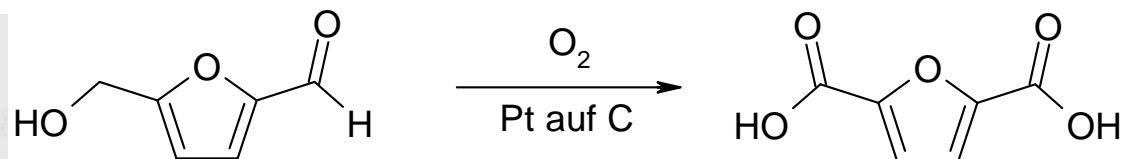
HMF  
(raw material)

residue



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

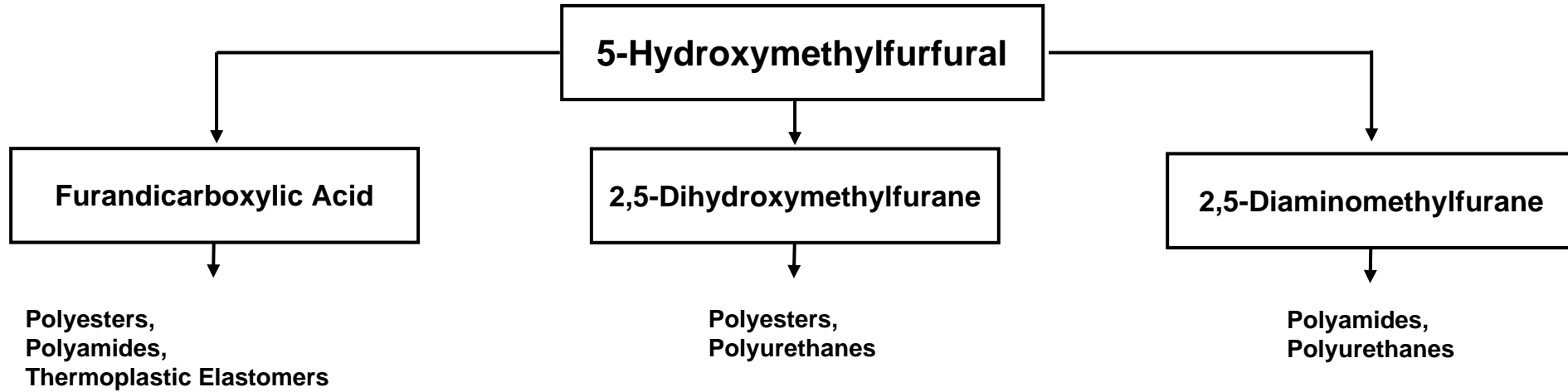
## ICT Synthesis of 2,5-Furandicarboxylic acid (FDCA)



Oxidation of 5-Hydroxymethylfurfural in water

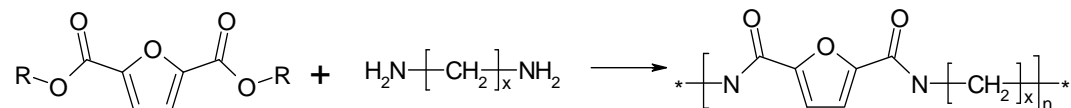
Batch size max. 2,5 kg 5-HMF

Yield FDCA > 98 %

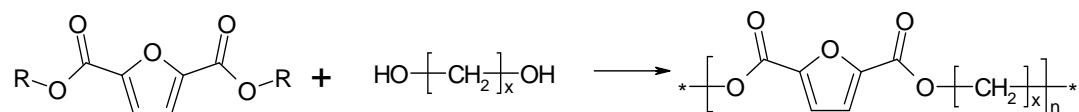


## Biopolymers based on Furanedicarboxylic acid

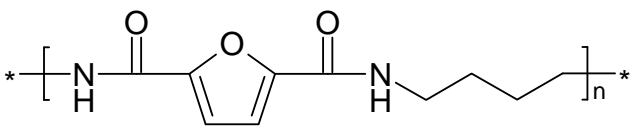
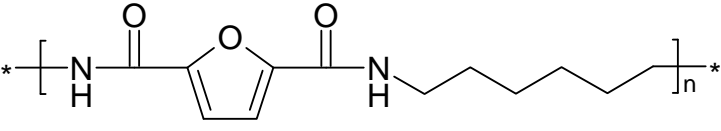
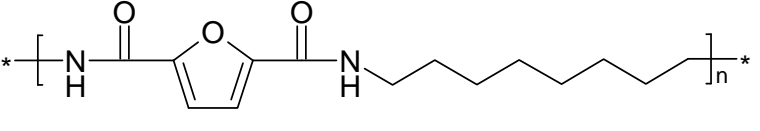
### Polyamides



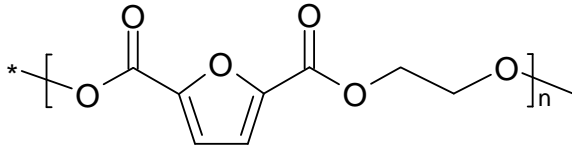
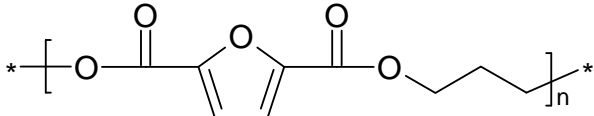
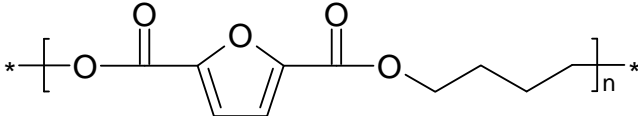
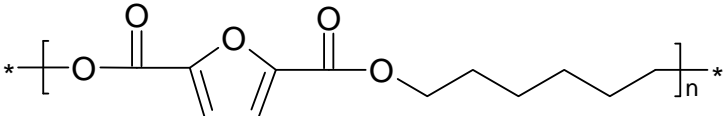
### Polyesters



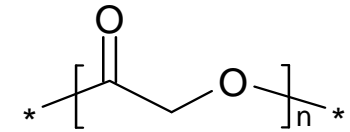
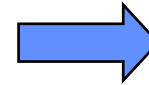
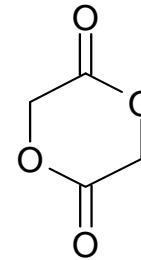
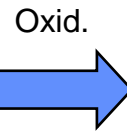
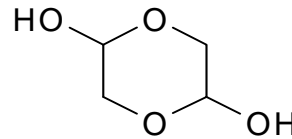
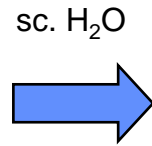
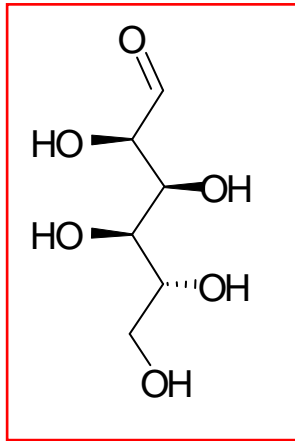
## Thermal properties polyamides of glucose based polyamides

 $* \left[ \text{NH} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_4 - \right]_n *$	<p><math>T_m = 250 \text{ }^\circ\text{C}</math></p>
 $* \left[ \text{NH} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_6 - \right]_n *$	<p><math>T_m = 175 \text{ }^\circ\text{C}</math> Fibres</p>
 $* \left[ \text{NH} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{NH} - (\text{CH}_2)_{10} - \right]_n *$	<p><math>T_m = 125 \text{ }^\circ\text{C}</math></p>

## Thermal properties of glucose based polyesters

	<p><math>T_m = 205-210\text{ }^\circ\text{C}</math> Fibres from melt</p>
	<p><math>T_m = 120\text{ }^\circ\text{C}</math></p>
	<p><math>T_m = 163 - 165\text{ }^\circ\text{C}</math></p>
	<p><math>T_m = 143 - 146\text{ }^\circ\text{C}</math></p>

## Glucose



Polyester	T <sub>m</sub> (°C)
Poly(3HB)	178
Poly(3HV)	110
Poly(L-lactid)	185
Poly(glycolid)	223-228

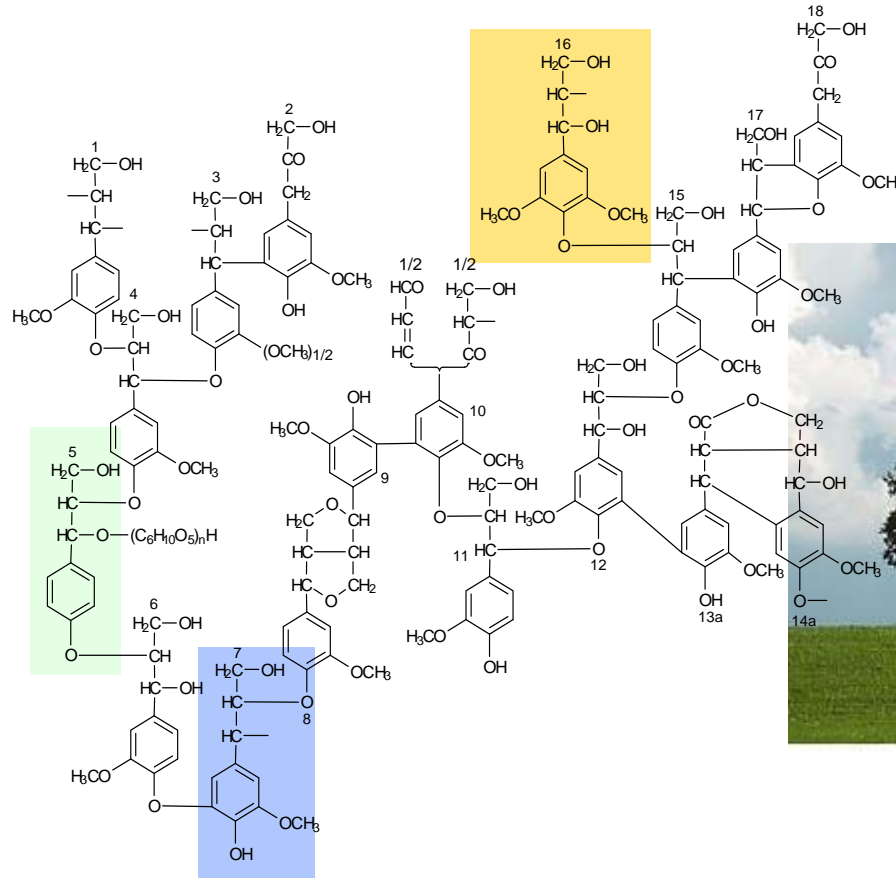




# Lignin separation and transformation



# Lignin



## Lignin release from wood

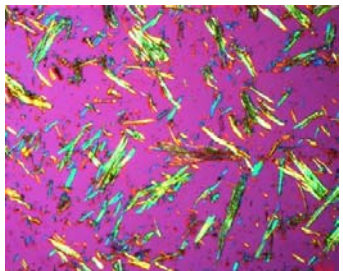
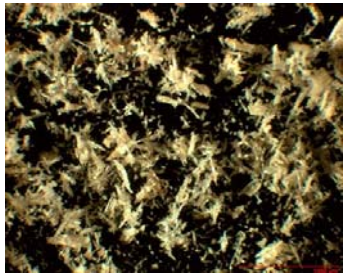
- Sulfate or sulfite process
- Acid wood digestion
- Ionic liquids
- Hydrothermal Treatment (Aquasolv-Process)
- **Organosolv-process (Ethanol, ...)**
- Enzymatic cleavage (+ Fraunhofer IGB)



Lignin production:

## The ORGANOSOLV-Process

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



Autoclave (13 L)



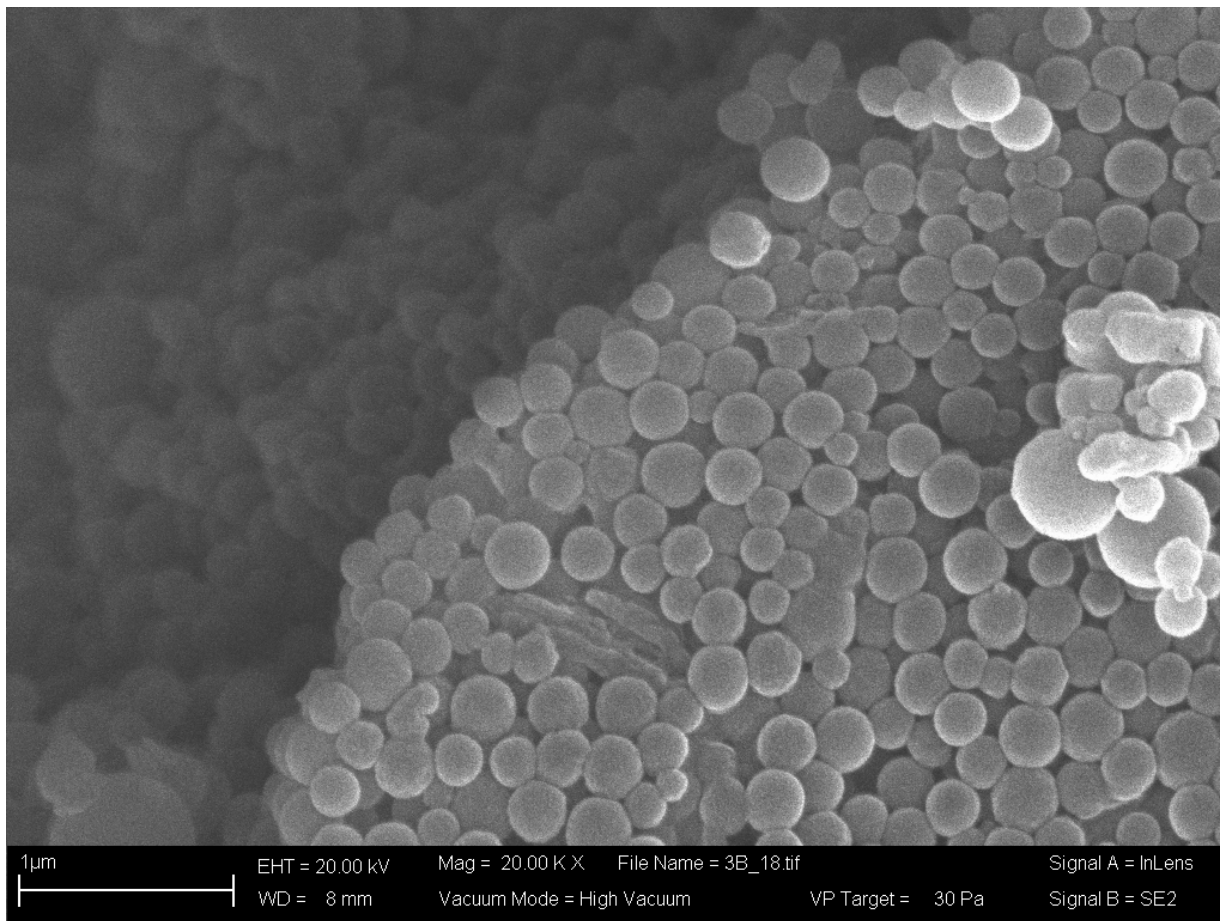
Separation  
Solvent Regeneration



Beech wood

**Nanosized**

**Organosolv-Lignin**

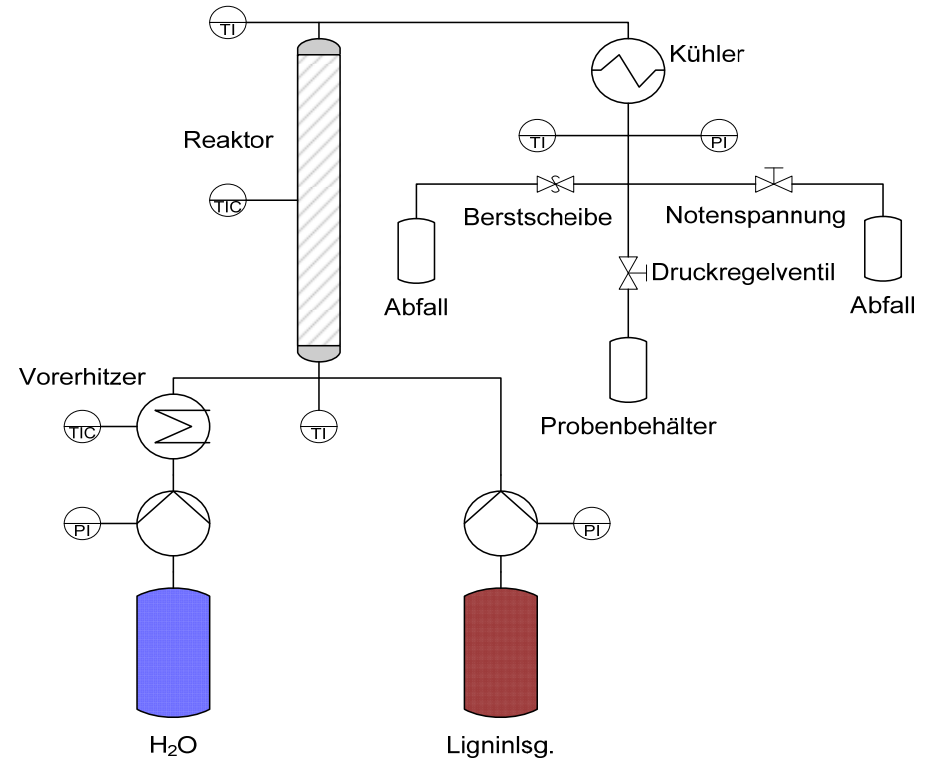


# Lignin Transformation: Hydrothermolysis



## Process parameter

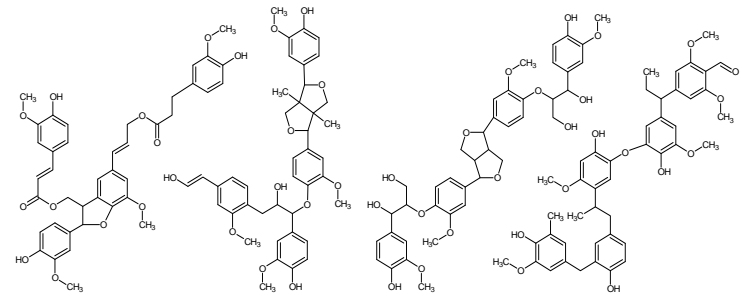
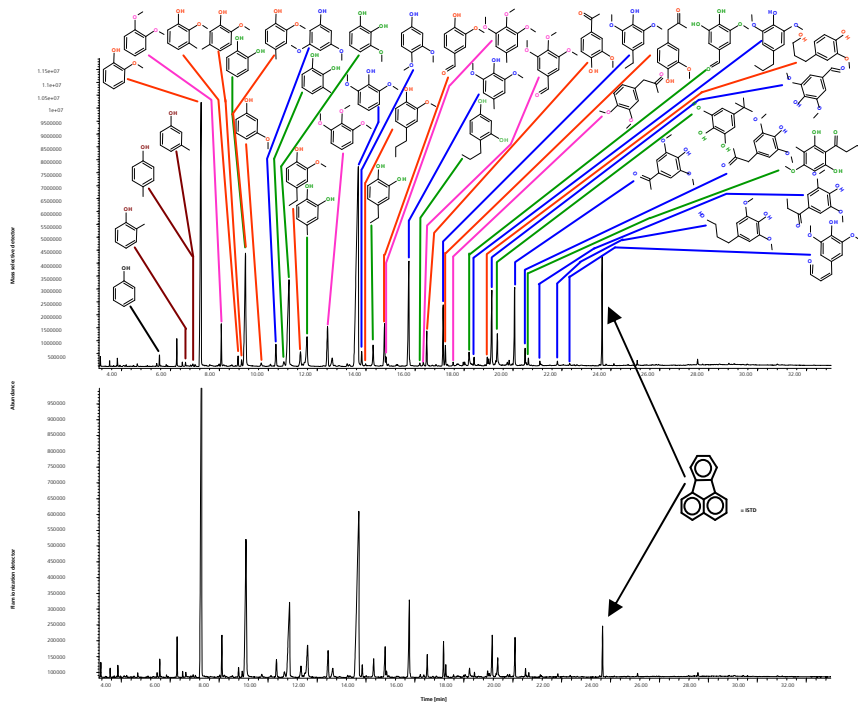
max. temperature:	400°C
max. pressure:	250 bar
max. flow rate:	7,3 L/h
Reactor length:	8- 12 m
Reactor diameter:	9 mm
Residence time:	15 min



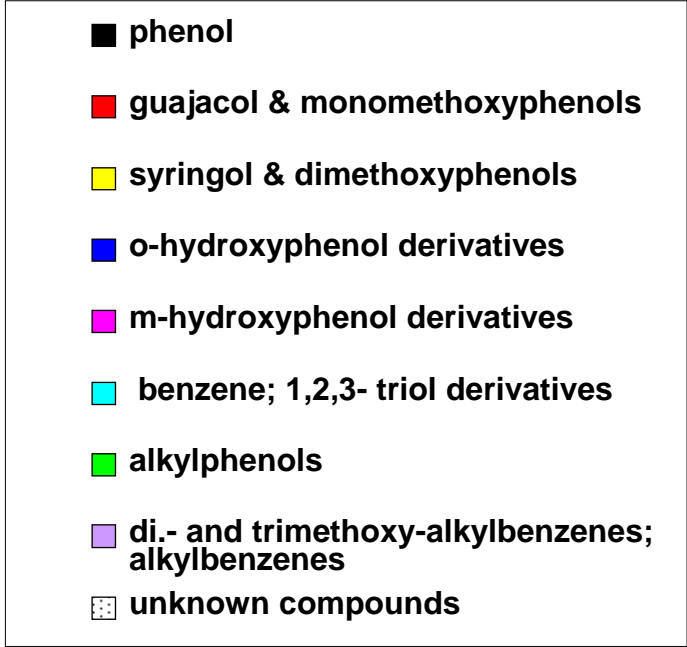
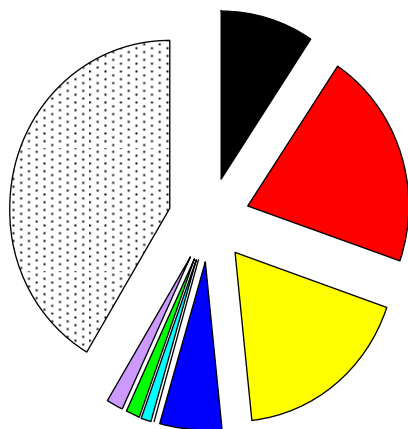
# Chemical composition of the products

## Fraction of oligomers

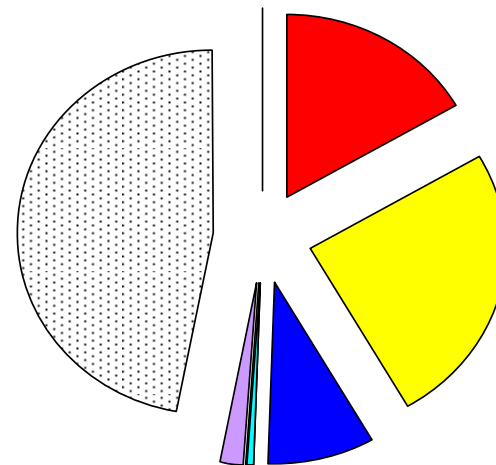
Fraction of monomers



**Poplar lignin**



**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)

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**Thank you for your attention !**