

Separation of lignin and hemicelluloses from alkaline process liquors

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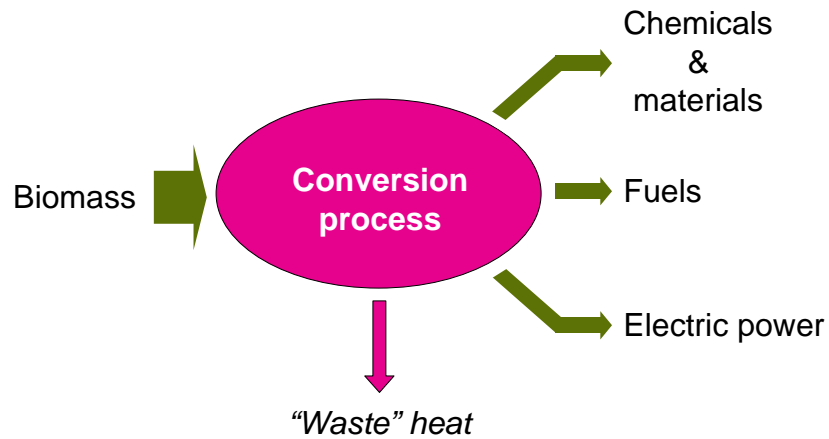
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Outline

- Background & motives
- Experimental
- Results & conclusions
 - Sequential precipitation of lignin
 - Separation of xylan
- Aspects on industrial implementation
- Acknowledgements

A biorefinery system



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Examples on process efficiencies¹

Concept	Main products	Process efficiency ² , %
Rankine power	Electricity	33
CHP ³	Electricity and district heat	90-95
Ethanol + Rankine power	Ethanol, electricity	61
EtOH+FT(recycle)+CH ₄	Ethanol, FT-fuels	80
Conventional petroleum refining	Fuel, petrochem., other	85

¹ As predicted in their mature form. Selected data from: [Laser et al., Biofpr. 3:247-270\(2009\)](#)

² 100· Produced energy (fuel, power, feed) / NHV_{fuel}. Biomass (or fuel) = switchgrass.

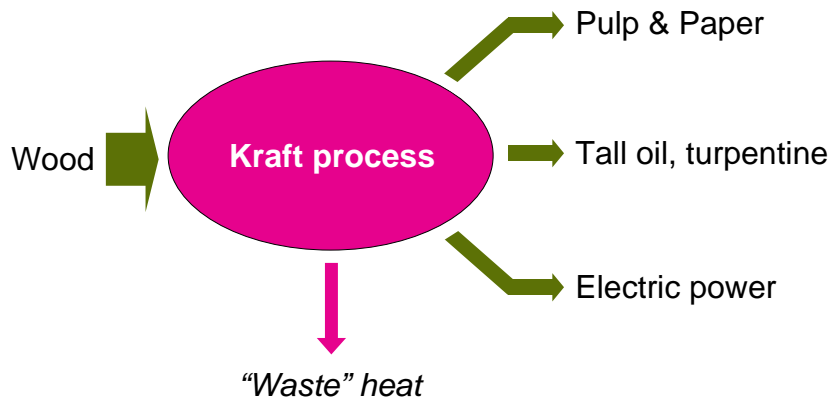
³ E.g. Alvarez 2003.

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Kraft pulp mill platform - today

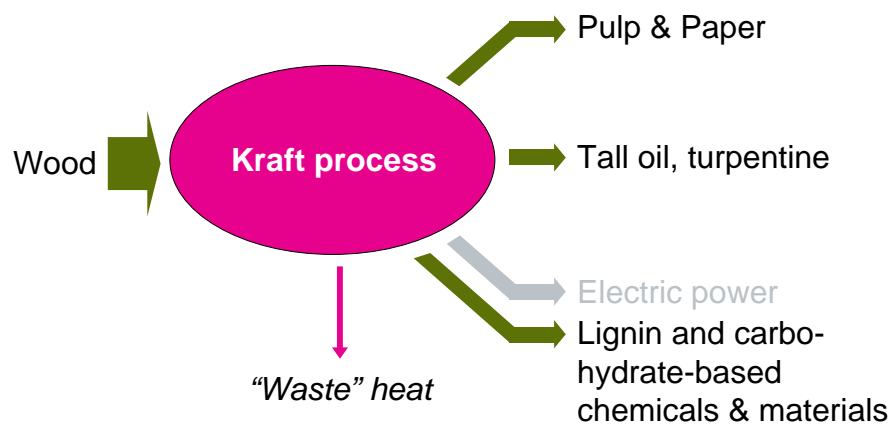


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Kraft pulp mill platform – tomorrow?



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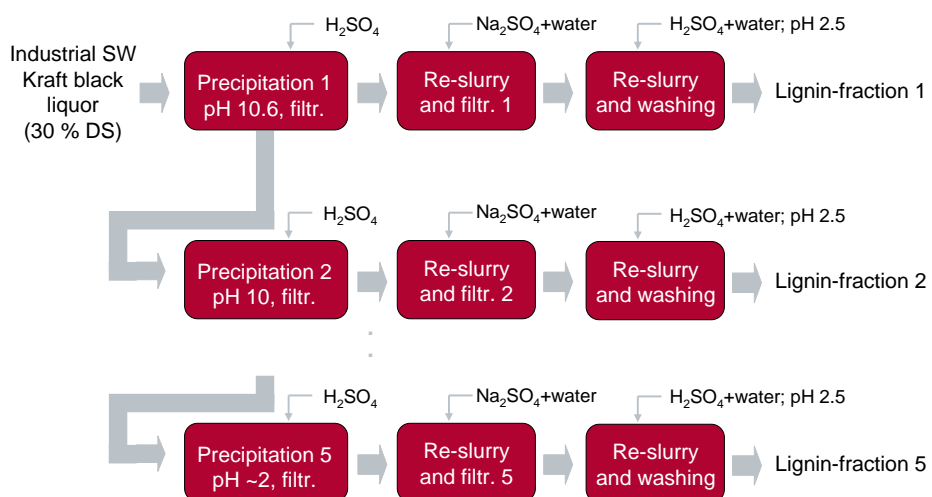
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Fractionation of lignin *via* sequential precipitation

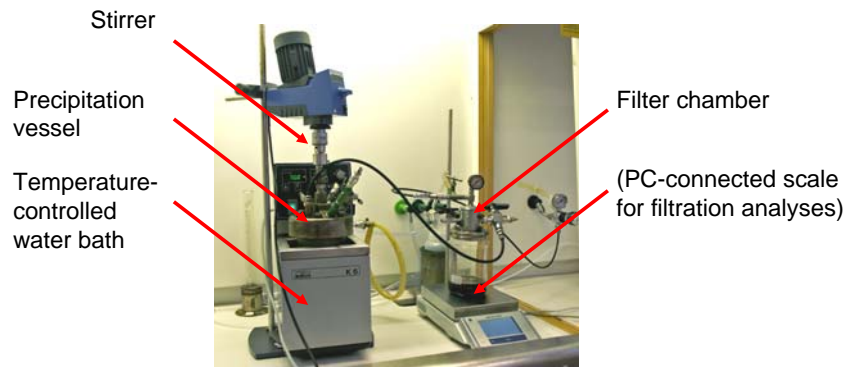


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Equipment for precipitation and filtration



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Extraction of birch wood chips and ultrafiltration

Alkaline extraction



Volume: 300 L
Wood: ca. 25 kg
Conditions: ~ 50 gNaOH/l, 120°C, 110 min.

Ultrafiltration



Membranes: Ceramic, 5 and 15 kDa
TMP: 3.5 bar (5kDa) 1.5 bar (15kDa)

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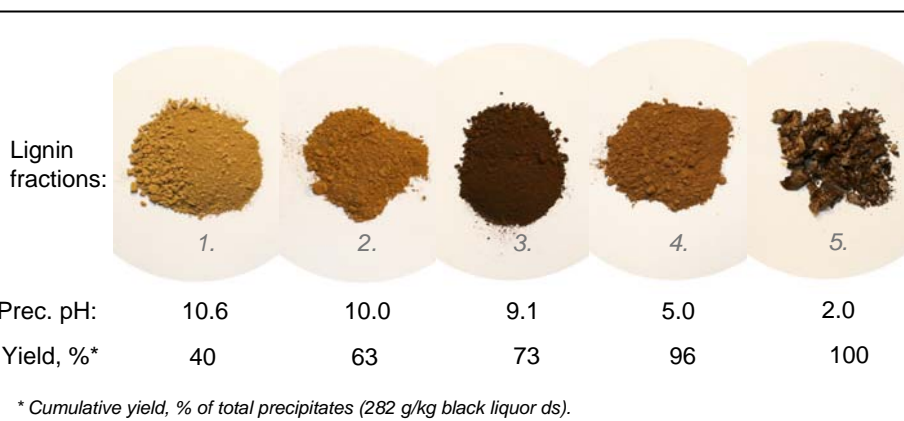
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Visuals and precipitation yields



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Chemical compositions

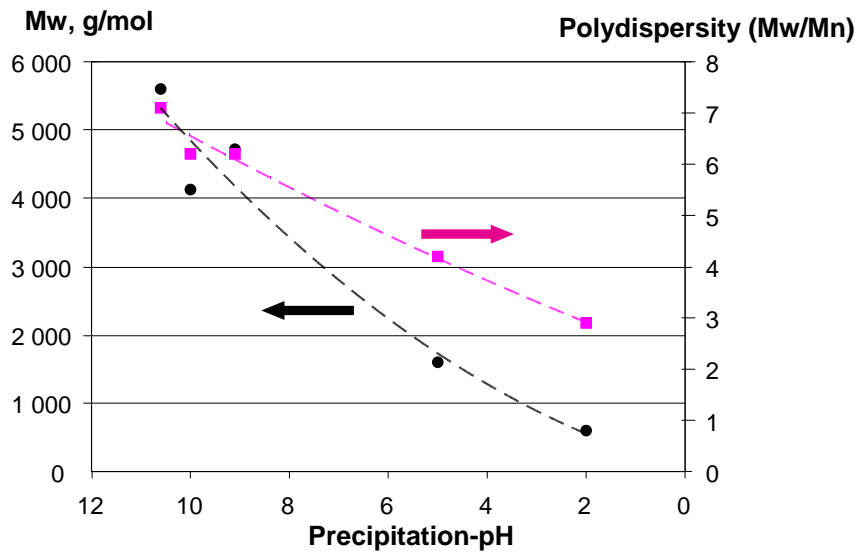
Fraction, precipitation pH	10.6	10.0	9.1	5.0	2.0
Isolated solids, g/kg blds	110	70	25	65	12
Klason lignin, w-%	93.6	91.2	95.6	87.7	45.8
Acid soluble lignin, w-%	3.6	6.6	4.0	9.6	33.5
Total lignin, w-%	97.2	97.8	99.6	97.3	79.3
Total carbohydrates, w-%	1.6	1.1	1.2	0.9	2.6
Na, w-%	0.3	0.4	0.4	0.2	0.4
Sum of lignin, carboh. and Na, w-%	99.1	99.3	101.2	98.4	82.3

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Molecular mass distribution

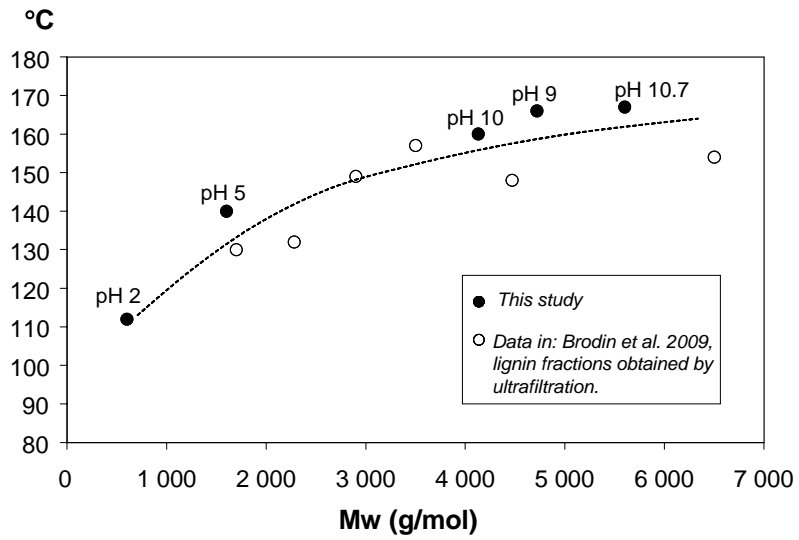


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Glass transition temperature



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Conclusions I: Lignin fractionation *via* sequential precipitation

- Successful method for precipitation and filtration of lignin fractions.

	Lignin fractions	
	10.6 > pH > 9.1	9.1 > pH > 2.0
Lignin yield	Higher	Lower
Molecular mass	Higher	Lower
Polydispersity	Higher	Lower
Glass transition temperature	Higher	Lower

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Basic data on alkaline extraction and ultrafiltration

Wood: Birch (*B. pendula*)

Conditions for alkaline extraction: 50 gNaOH/l, 12 l/kg wood, 120°C, 110 min.

	Wood	Feed	Permeate ¹	Retentate ¹	Balance ² , %
Lignin, g/kg wood	(231)	42	31	10	3
Xylose, g/kg wood	(240)	26	1	25	1
Na, kg/kg wood	(~0)	0.3	0.3	~0	~ -2

¹ After diafiltration

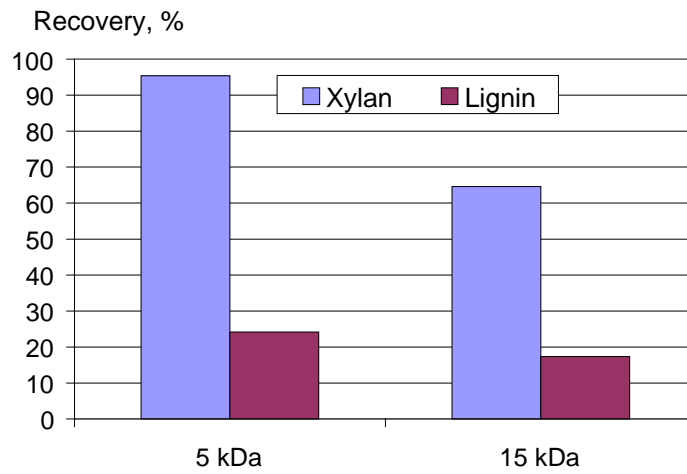
² $[(Feed) - (Permeate) - (Retentate)] / (Feed) \cdot 100$

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Ultrafiltration of alkaline extracts

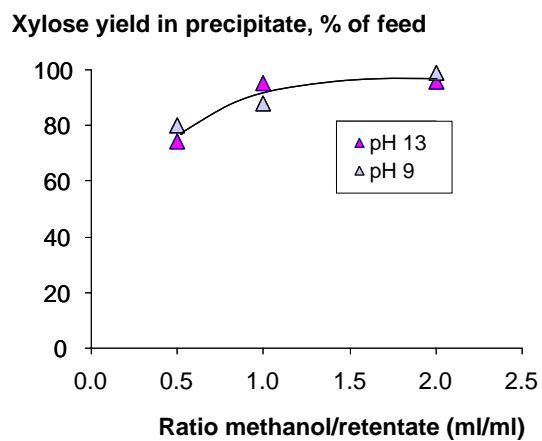


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Effect of acidulation on precipitation of xylose



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Conclusions II: Separation of xylan

- Ultrafiltration resulted in high retention of xylan and low retention of lignin
- Acidulation (pH 13 \Rightarrow 9) did not significantly affect yield of isolated xylan
- Increasing the alcohol:liquor ratio above 1 only marginally affected the xylan-yield

Aspects on implementations in the Kraft pulp mill

	Lignin fractions	Xylan
Product applications	CF, adhesives, other?	Component in packaging barriers, fibre additive
Separation process	Multiple filtrations, capital & operating costs?	Alcohol recovery and make-up, capital & operating costs, xylan quality
Integration	Type of acid, Na/S-balance, energy balance	Position, energy balance, integration with LignoBoost?
Business models	New products - supplier of bulk raw materials or final products? New customers and competitors New sales management	

Acknowledgements

- Financing companies of the Biorefinery cluster: Alabama River Pulp, Aracruz Celulose, Kemira, Perstorp, Sappi, Stora Enso, Södra Cell, UPM-Kymmene and Weyerhaeuser.
- VINNOVA (The Swedish Governmental Agency for Innovation Systems)
- Therese Alm and Ann-Marie Olsson at Innventia AB for chemical and thermal analyses.