VTT Technical Research Centre of Finland

Organosolv cooking as a facile fractionation method for annual plants

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Introduction of novel organosolv LGF-Process

Fractionation of Biomass

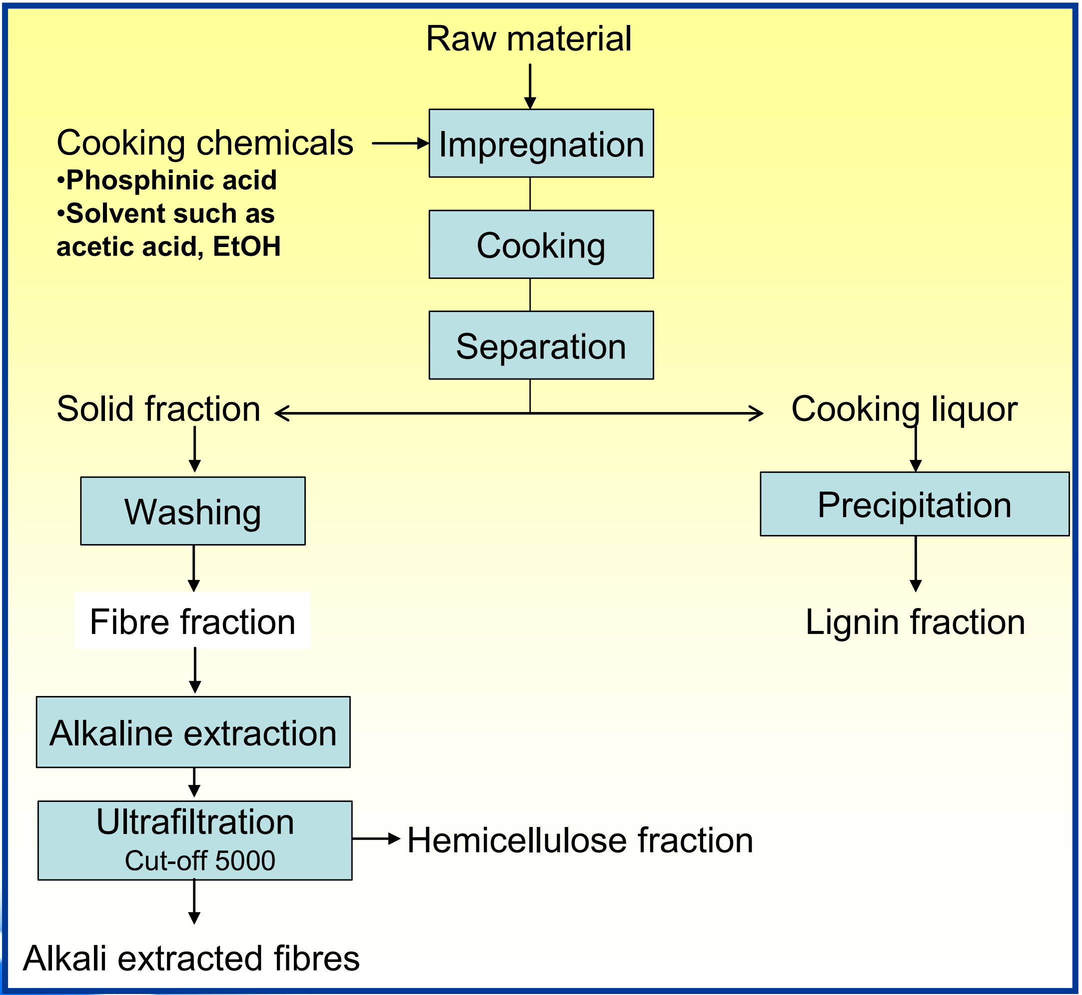


Figure 1. Schematic presentatio LGF pulping and subsequent fractionation processes

Fractionating reed canary grass by organsolv cooking

Table 1. Acetic acid/ H_3PO_2 cooking of reed canary grass

ACETIC ACID COOKING 26% moisture content of cooking liquor (CLQ)					
Sample	**t °C**	**Time (h)**	**CLQ used**	**Fiber yield %**	Hydrolysis-%
RA-1	**105**	**4**	**twice**	**57.4**	**59.3**
RA-2	**105**	**8**	**once**	**54**	**47.8**
RA-3	**105**	**13**	**once**	**51.0**	**47.4**
Sample	Post treatment of fiber with 1 M NaOH				
RA-1 Fiber	**80**	4		38	**100**

Symbols CLQ = Cooking liquor,
RA = Acetic acid cooking

- It is possible to use the same cooking liquor more than once (improved solid/liquid ratio)
- Complete hydrolysis of fiber is achievable when cooking is combined with alkaline extraction of fib

Fractionating reed canary grass by organsolv cooking

Table 2. Ethanol/ H_3PO_2 cooking of reed canary grass (R)

ETHANOL COOKING, 20% moisture content of cooking liquor (CLQ)					
Sample	**t °C**	**Time (h)**	**CLQ used**	**Fiber yield**	
RE-4	**79**	**30**	**twice**	**73.5**	**75.8**
Sample	Post treatment of fiber with 1M NaOH				
RE-4 Fiber	**80**	**4**		**57**	**100**

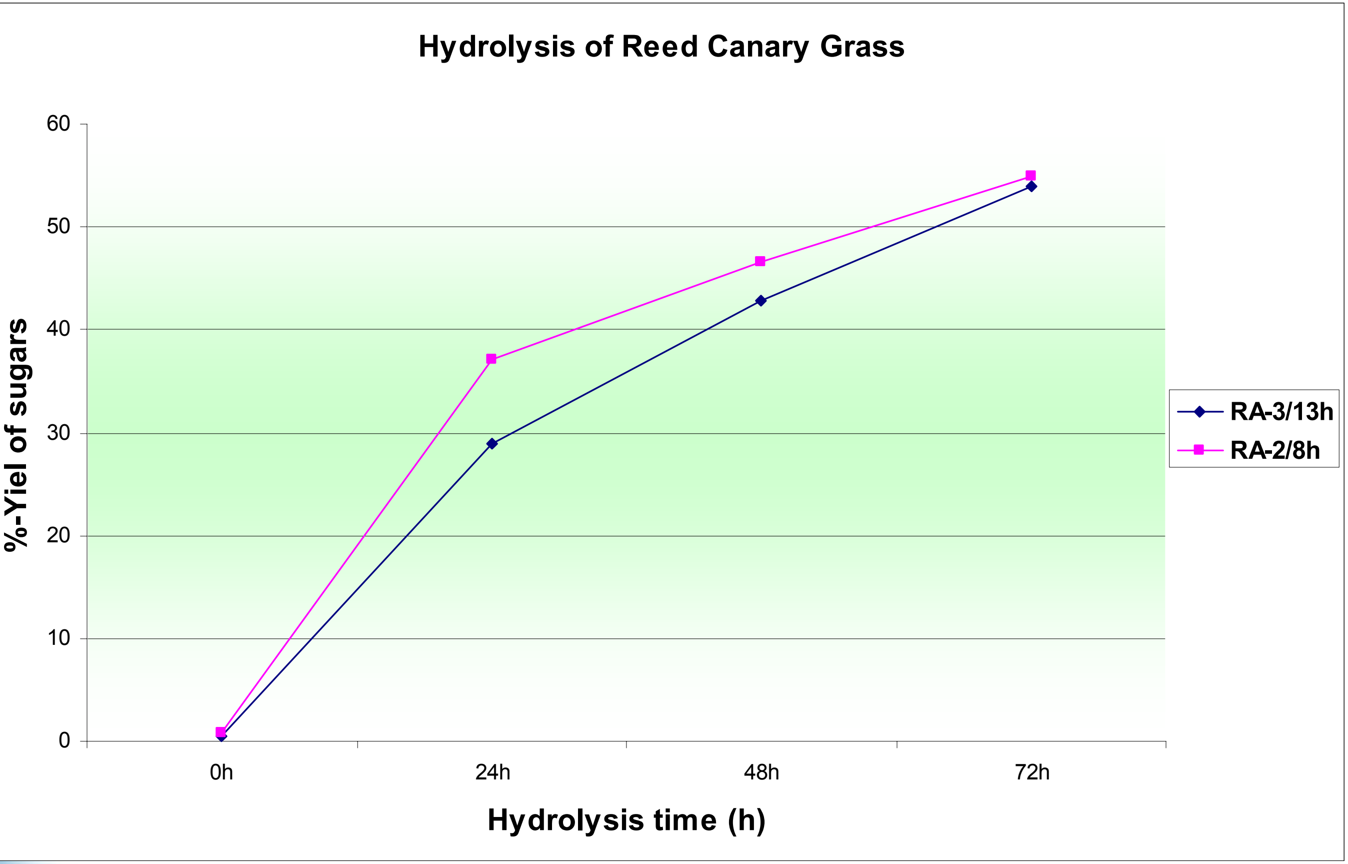
Symbols: CLQ: Cooking liquor, RE = Ethanol cooking

Table 3. Yields of isolated lignin from different cooking processes

Sample	Cooking liquid	**Lignin yield % (as isolated)**
RA-1	Acetic acid	**13.4**
RA-2	Acetic acid	**21.0**
RA-3	Acetic acid	**11.0**
RE-4	**Ethanol**	**7.0**

Very mild reaction conditio
can be used for partial remo
of lignin (i.e. boiling ethanc

Enzymatic hydrolysis rate of LGF treated biomass
Acetic acid cooking

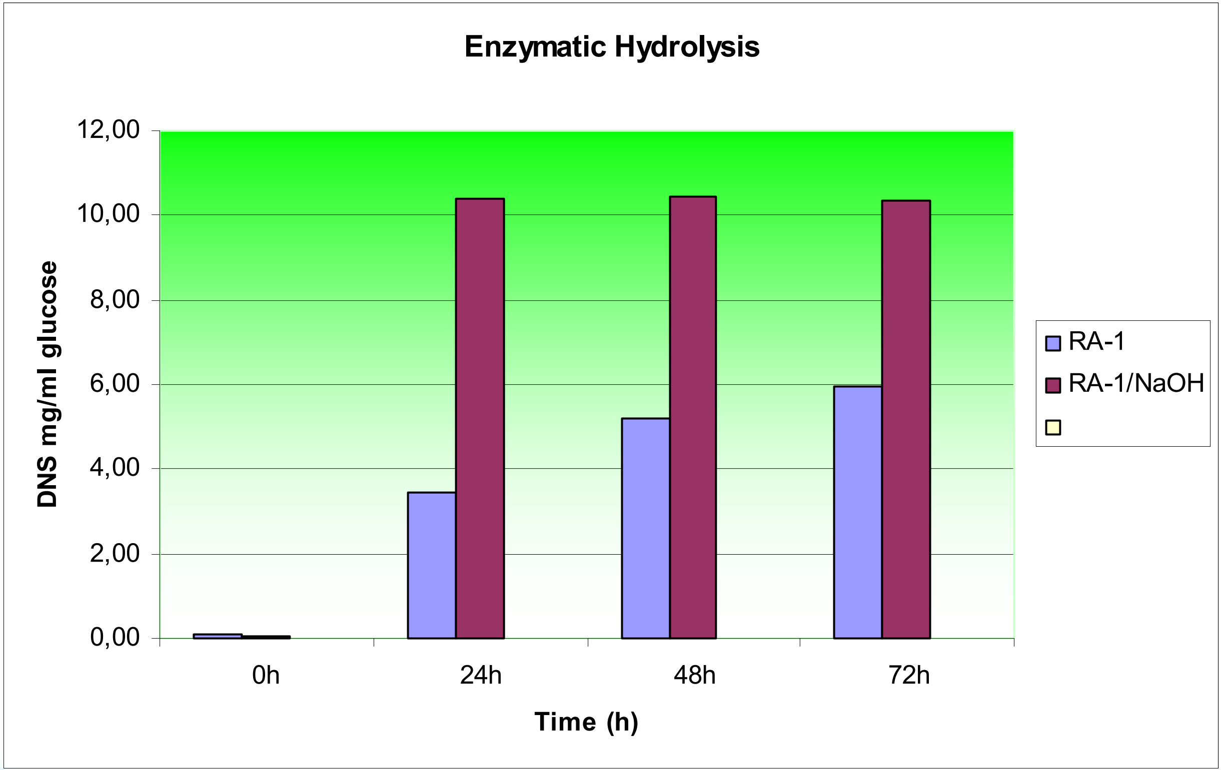


Long cooking time does not provide any advantage in hydrolysi

Cellulast 10 FPU/g
Novozyme 100 cat/g
10 g/l, pH5,45 °C

Figure 2. Effect of cooking time on hydrolysis rate

Enzymatic hydrolysis rate of LGF treated biomass
Acetic acid cooking
Fractionating

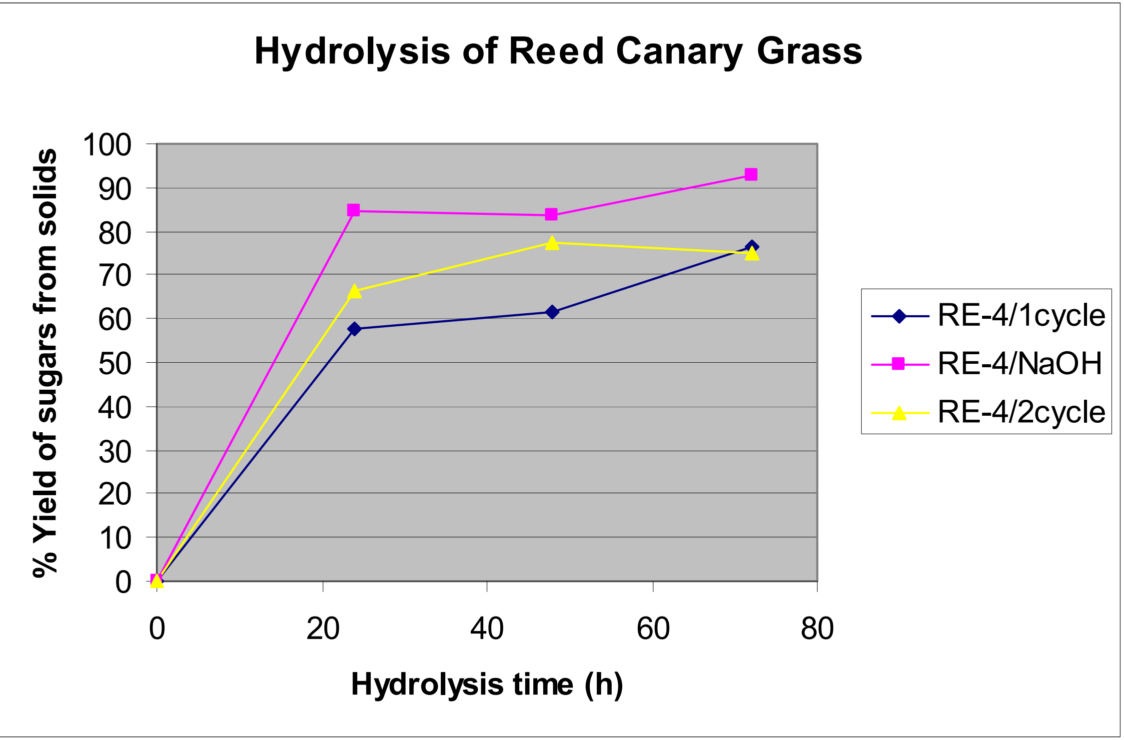


Complete hydrolysis of fiber i
achieved in 24 h after fraction
extraction with alkali

Probably alkali removes enzy
inhibiting components from fi
(e.g hemicellulose and/or resi
lignin)

Figure 3 Promoting hydrolysis rate of LGF fiber by alkaline extraction

Enzymatic hydrolysis rate of LGF treated biomass
Ethanol cooking
Fractionating

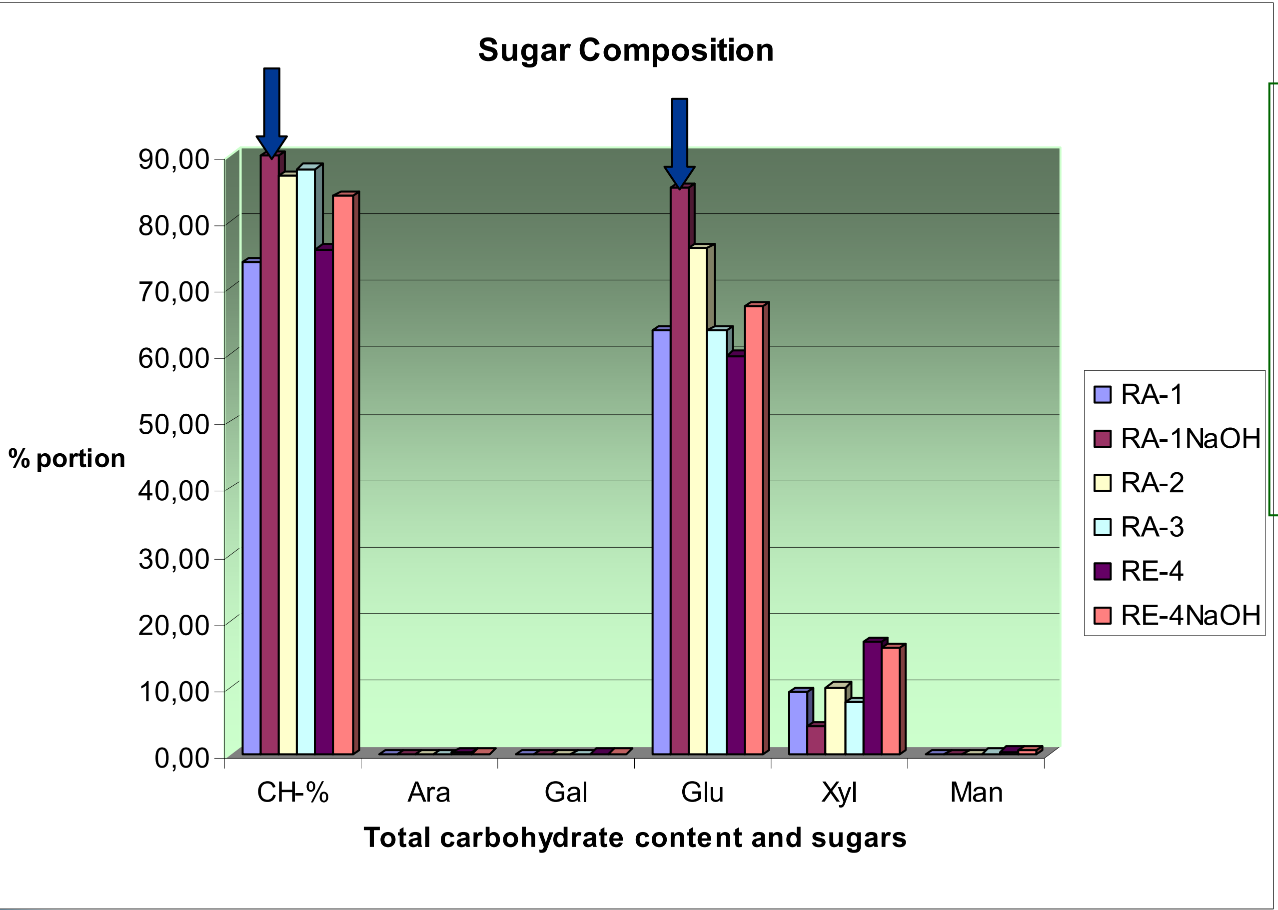


Re-usage of cooking liquor
Dos not have significant
effect on hydrolysis result

Alkaline extraction increased
significantly the hydrolysis rate

Figure 4. Results from ethanol LGF cooking. Recycling of cooking liquor and removal of enzyme inhibiting components.

Carbohydrate content of fiber (reed canary grass) and distribution of individual sugars



90 % carbohydrate content obtainable (RA: acetic acid coo
Mainly Glu and Xyl

Portion of hemicellulose is hig
Ethanol (RE) cooking, also de
amounts of arabinose, galactos
and mannose present.

NaOH extraction

Figure 5. Results from carbohydrate analysis. CH = % amount of carbohydrates of solids. Distribution (%) of individual sugars in carbohydrate part

Characterization of reed canary grass lignin NMR and DSC Results

Table 4 Background phosphorus (origin from H_3P0_2)

Lignin	at 2 ppm mmol/g	at 10- 50 ppm mmol/g	Total P mmol/g
RA	0.006	0.56	0.57
RE	0.041	0.35	0.39

Table 5 Amount of OH group species (mmol/g) in reed canary grass dissolved lignin

Lignin	Aliphatic OH	Aromatic OH	S+C	G	H	Carboxylic acid	Total OH
RE	3.3	1.8	0.4	0.8	0.6	0.3	5.4
RA	1.9	2.4	0.8	0.9	0.7	0.6	4.9

S+C = syringyl +condensed phenolic units
G = Guaiacyl unit
H = para-hydroxyphenyl phenolic unit

Table 6. Thermoplasticity of lignin

Lignin	Glass transition / °C	
	1. cycle	2. cycle
RE	65	61
RA	104	102
Protobind 1000, Reference	107	105

Conclusions

In addition to ethanol production, LGF-method is potential for fractionating lignocellulose materials to technically useful biopolymers in good overall yield.

Thank you for your attention

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