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Bioproducts, Sciences & Engineering Laboratory

Pretreatment of lignocellulosic materials using wet explosion

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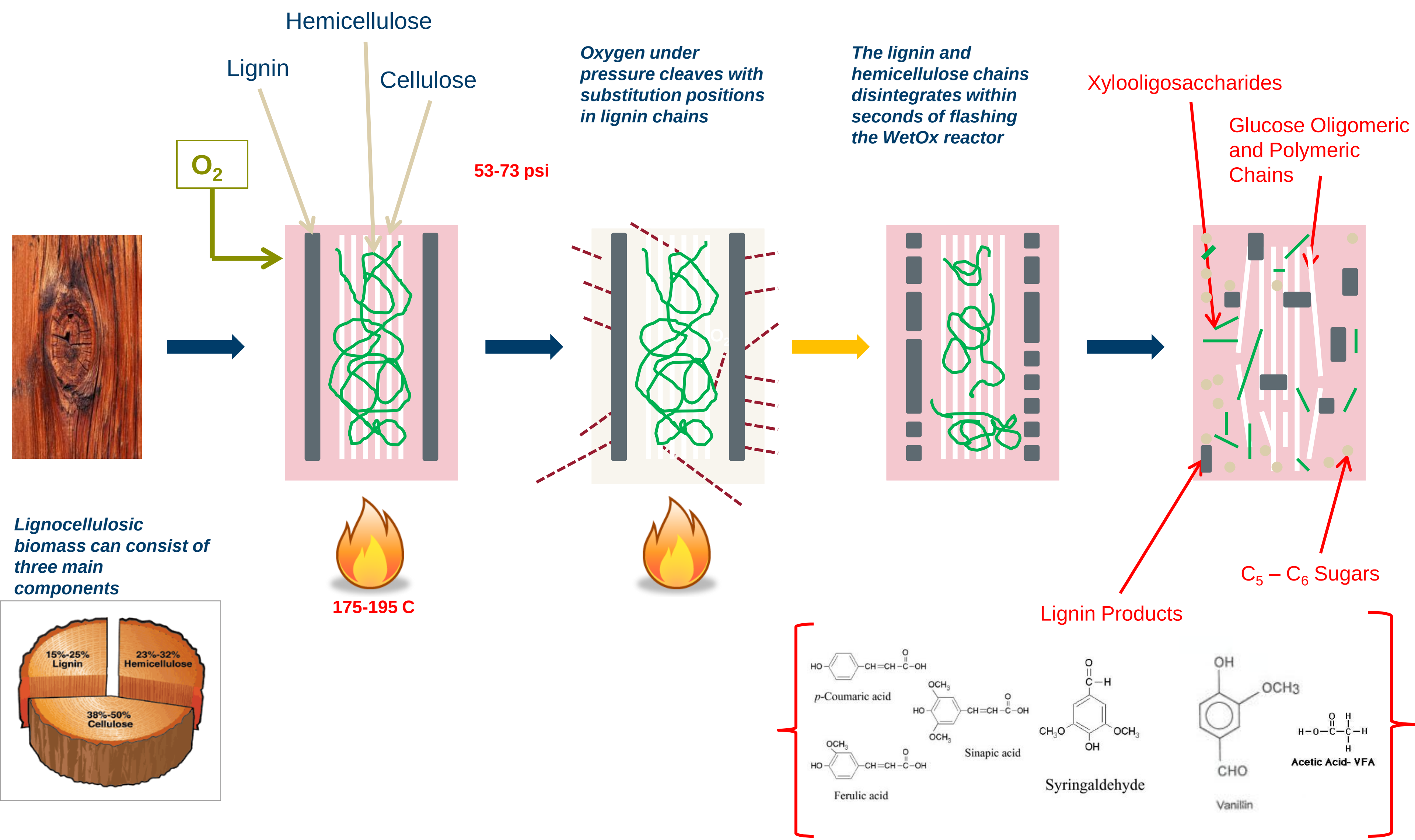
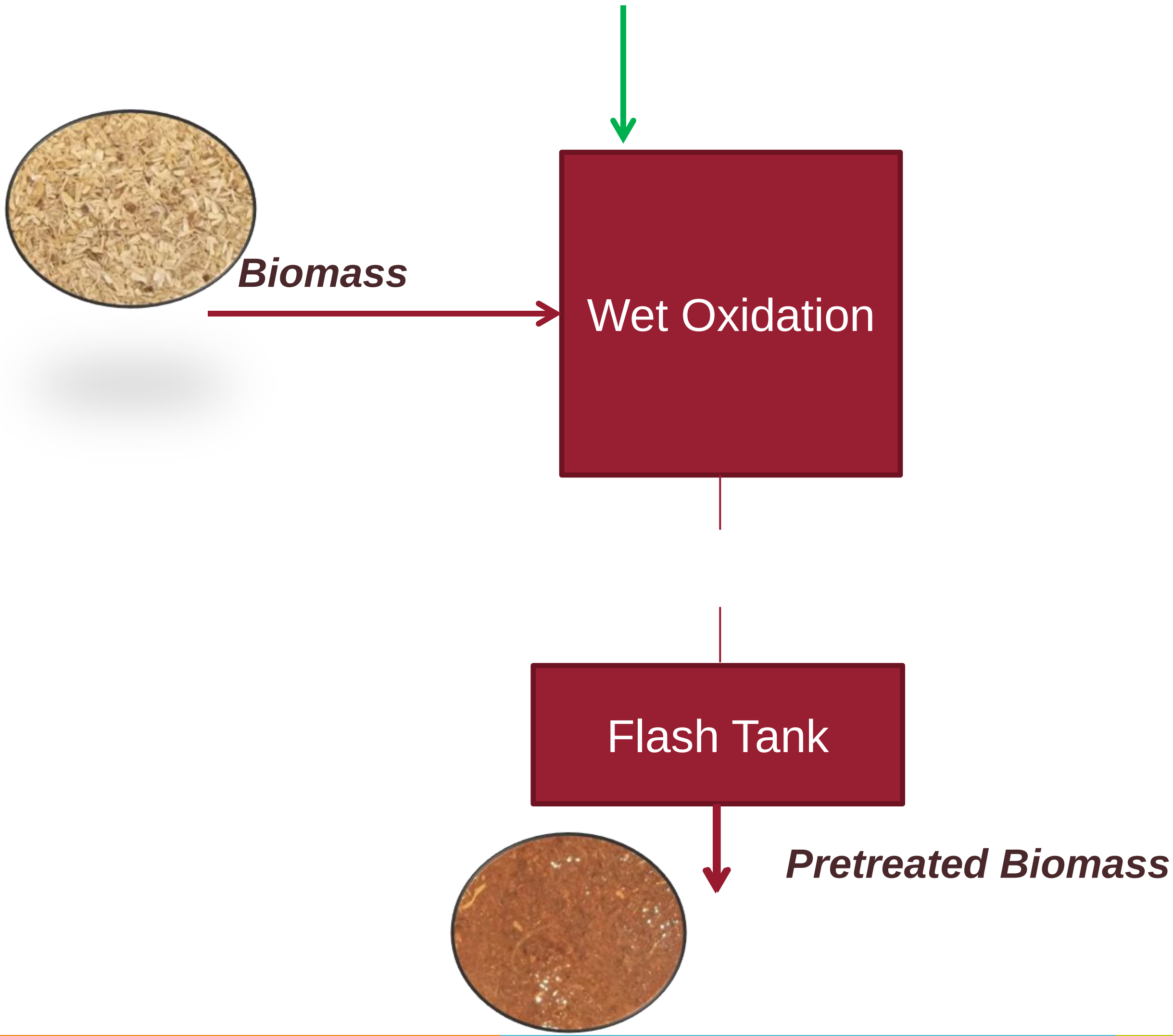
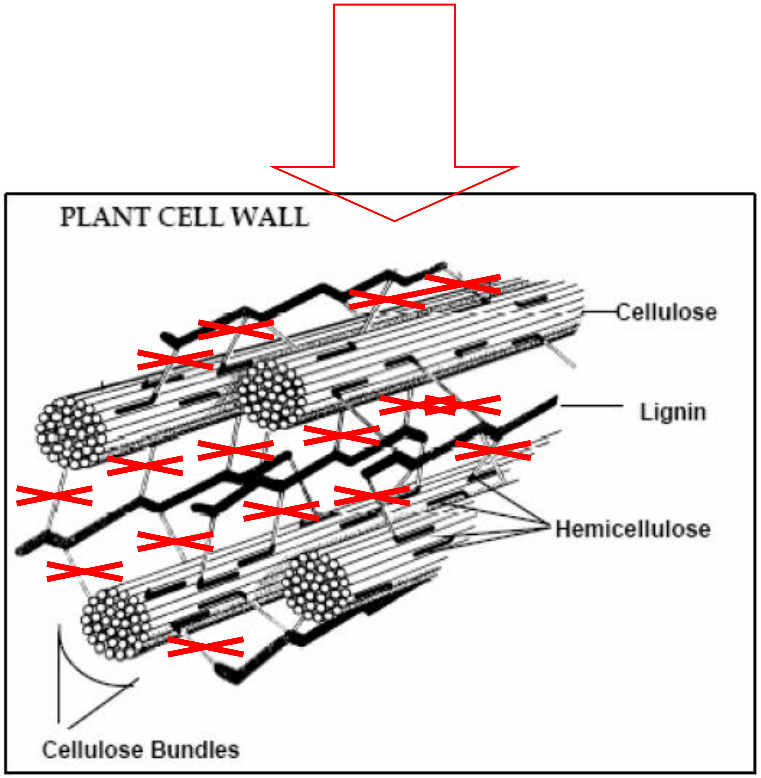
Wet Oxidation

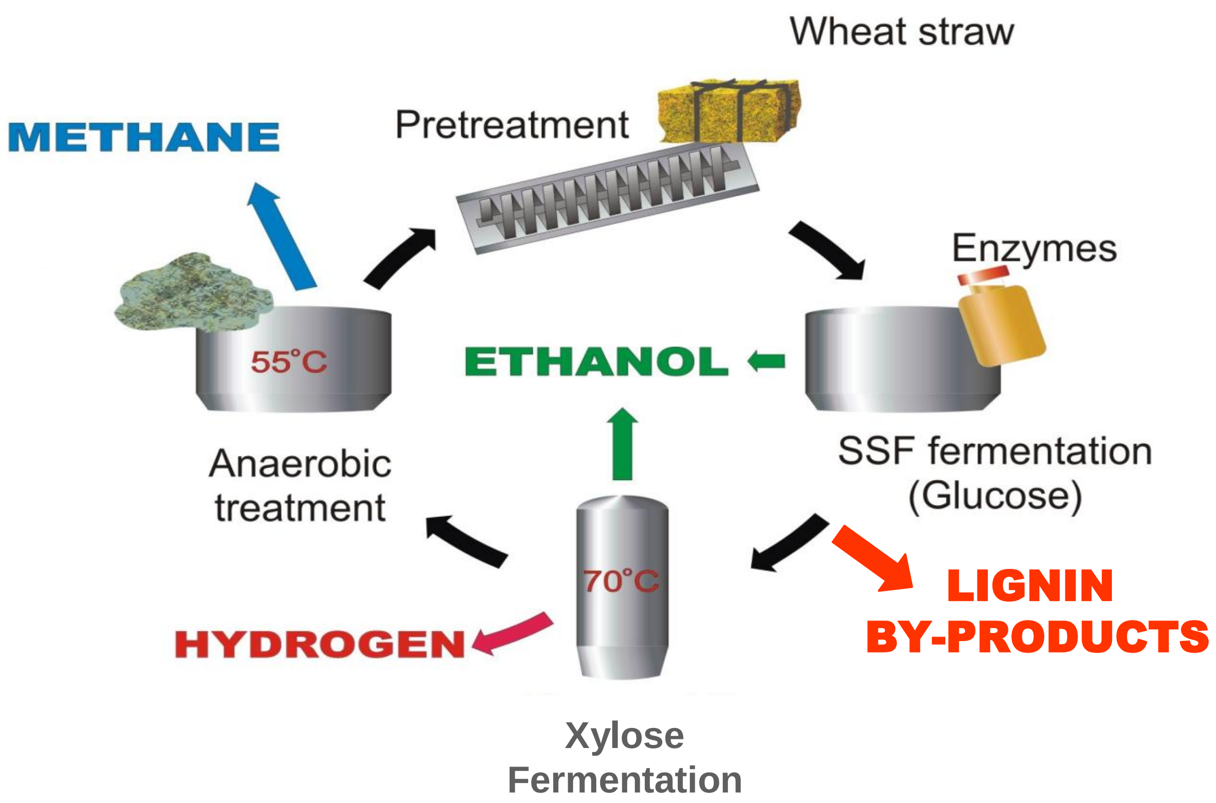


WET EXPLOSION PRETREATMENT




PLANT CELL WALL
Cellulose
Lignin
Hemicellulose
Cellulose Bundles

MaxiFuels biorefinery concept



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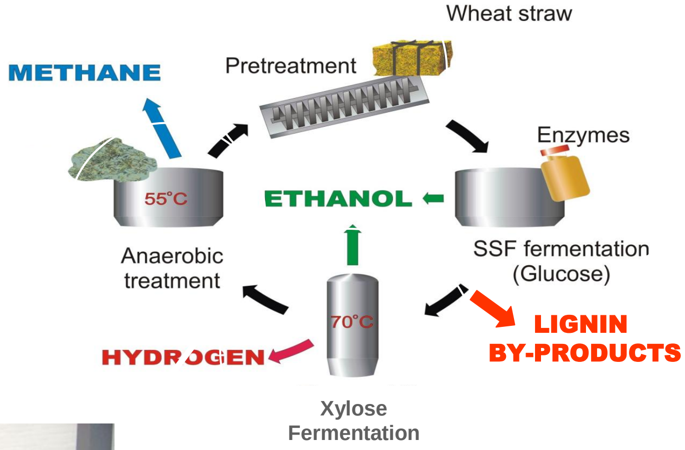
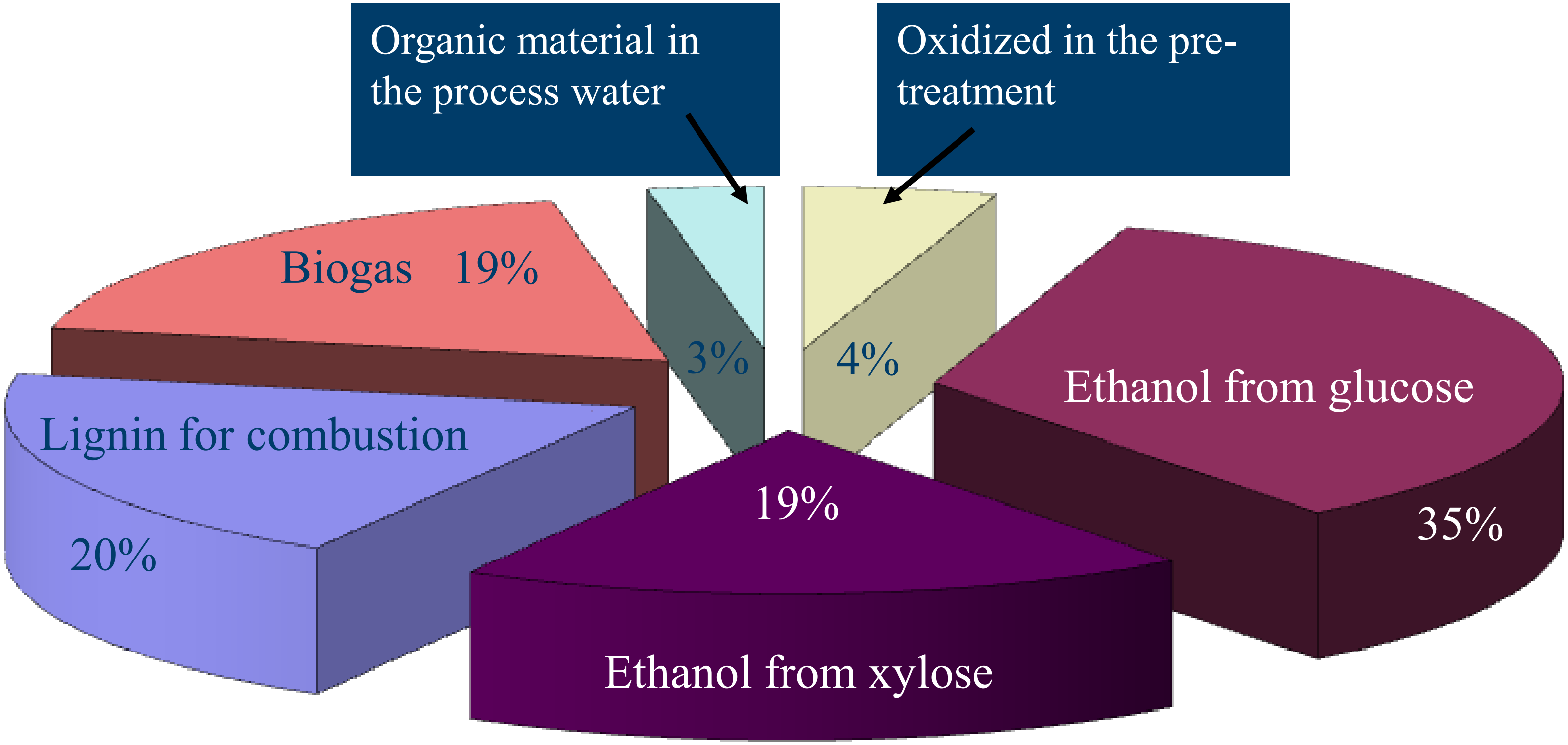
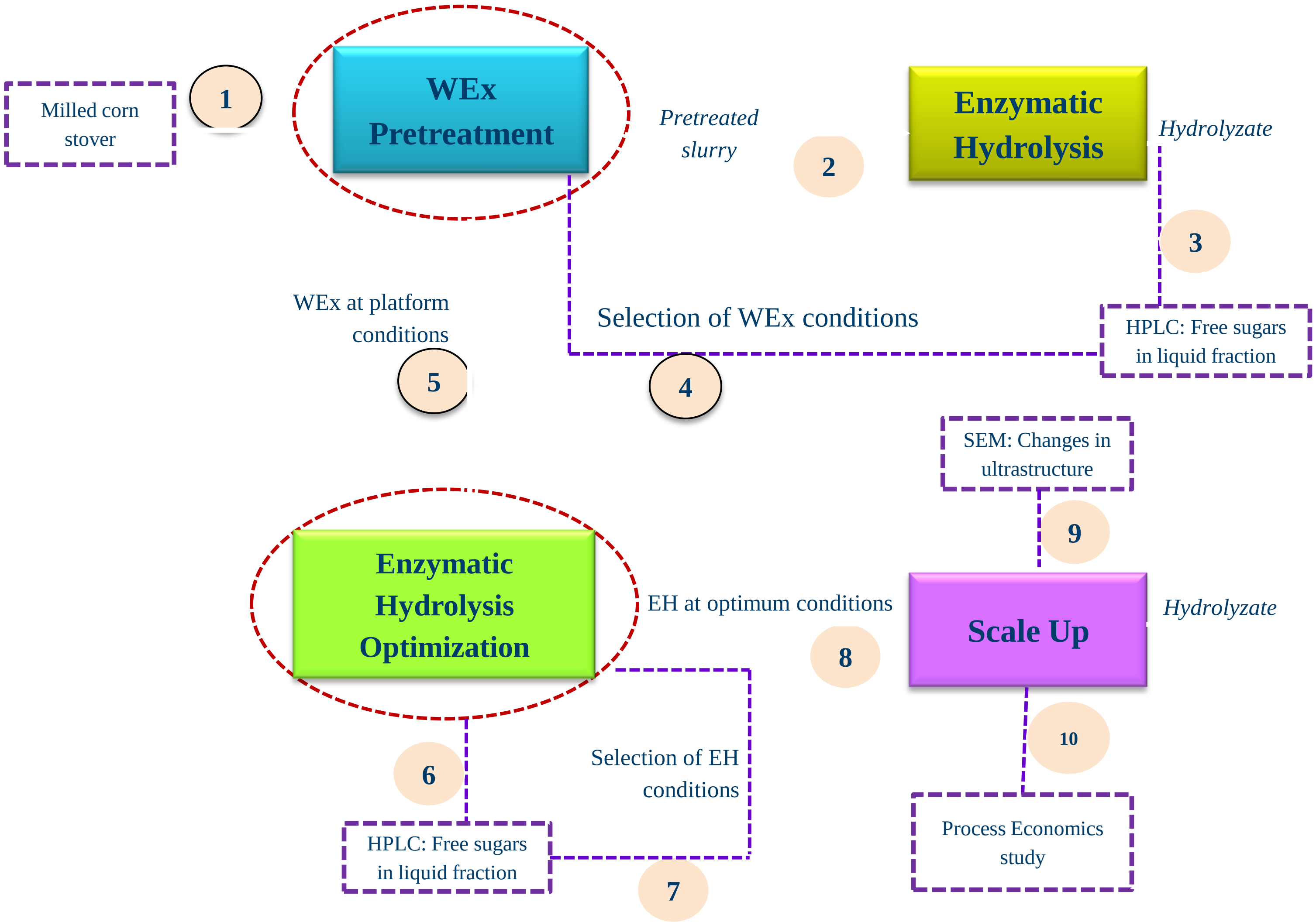
MaxiFuels biorefinery concept



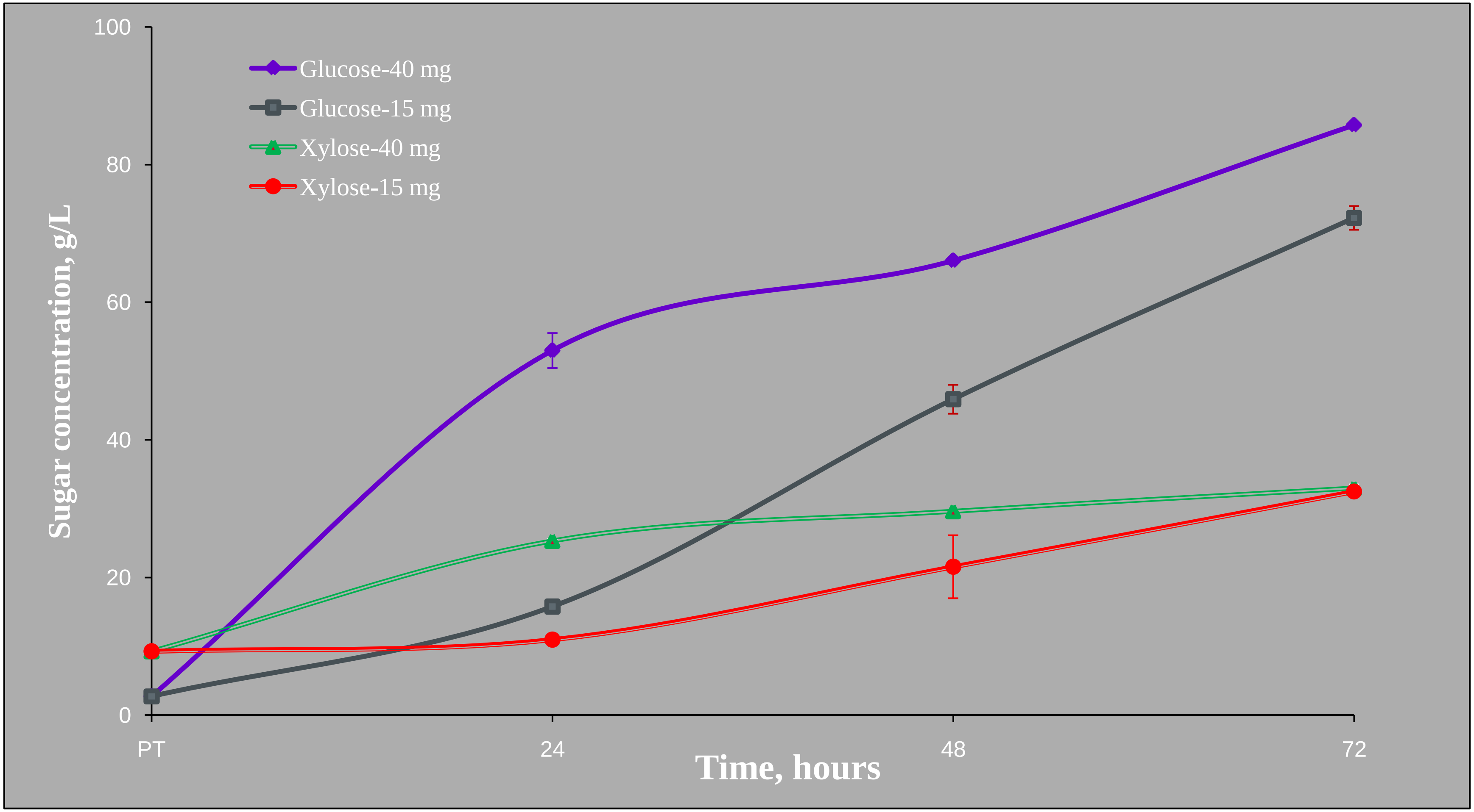
Optimized use of the biomass



Schematic of the study

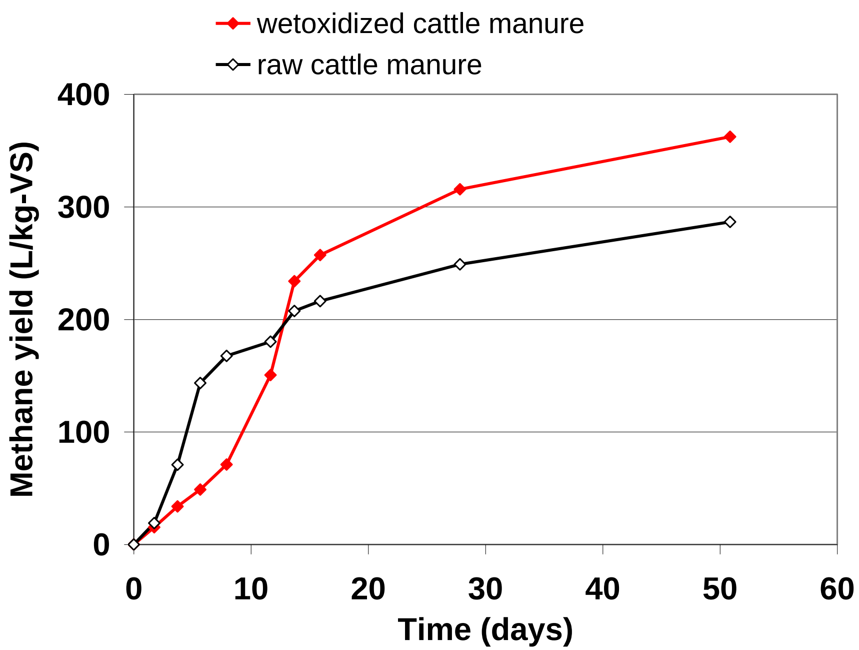


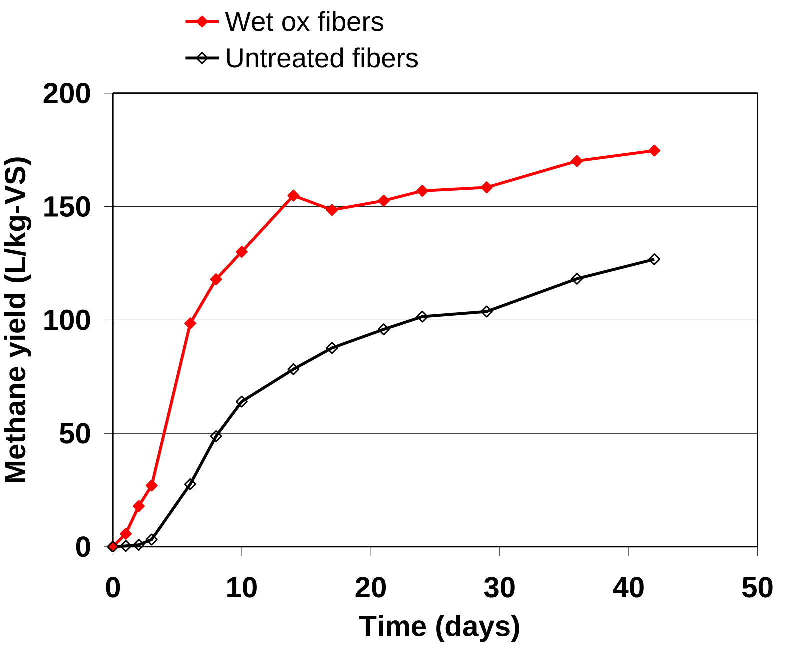
CS-8 WEx sample hydrolyzed @ 15 and 40 mg/g glucan

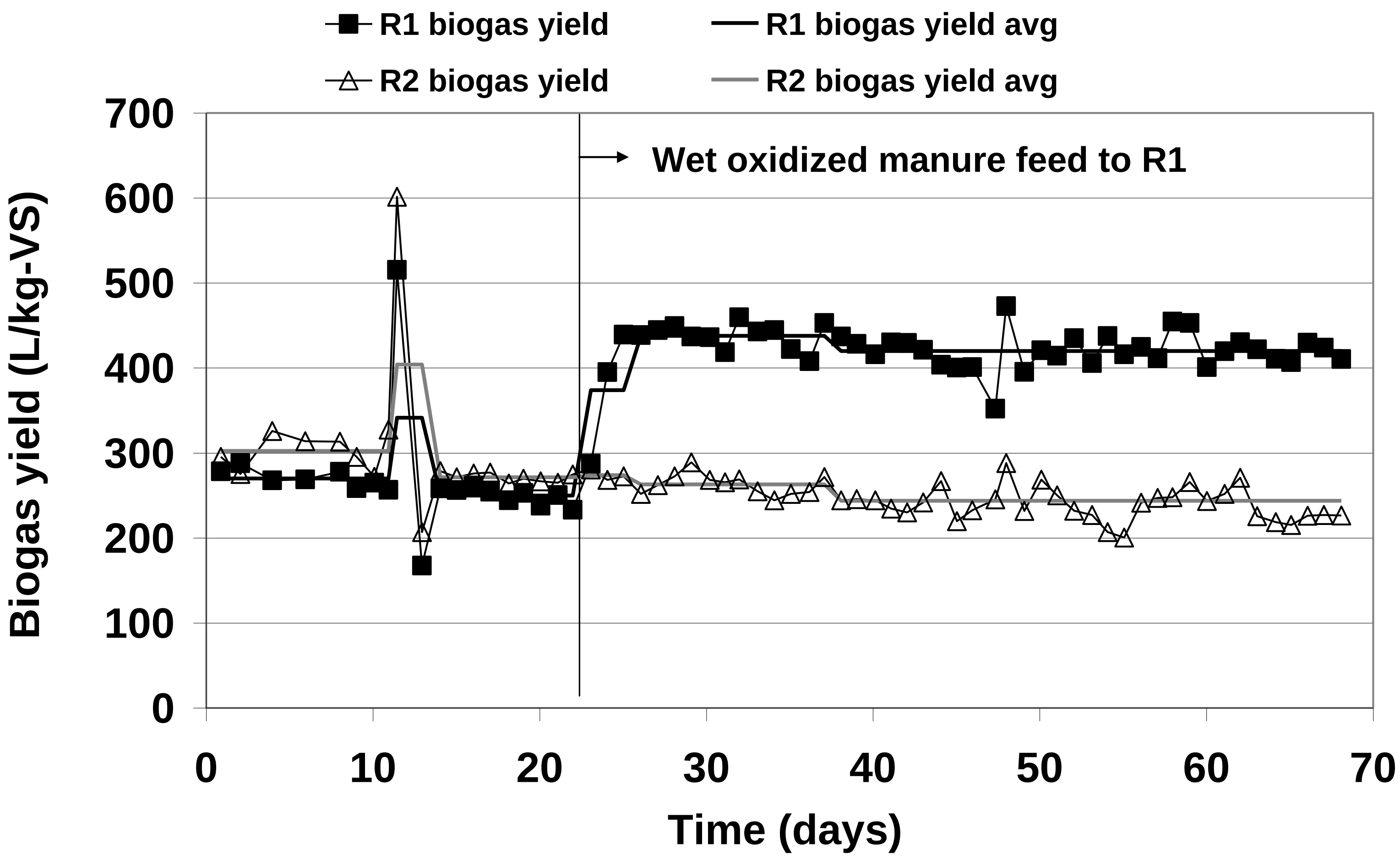


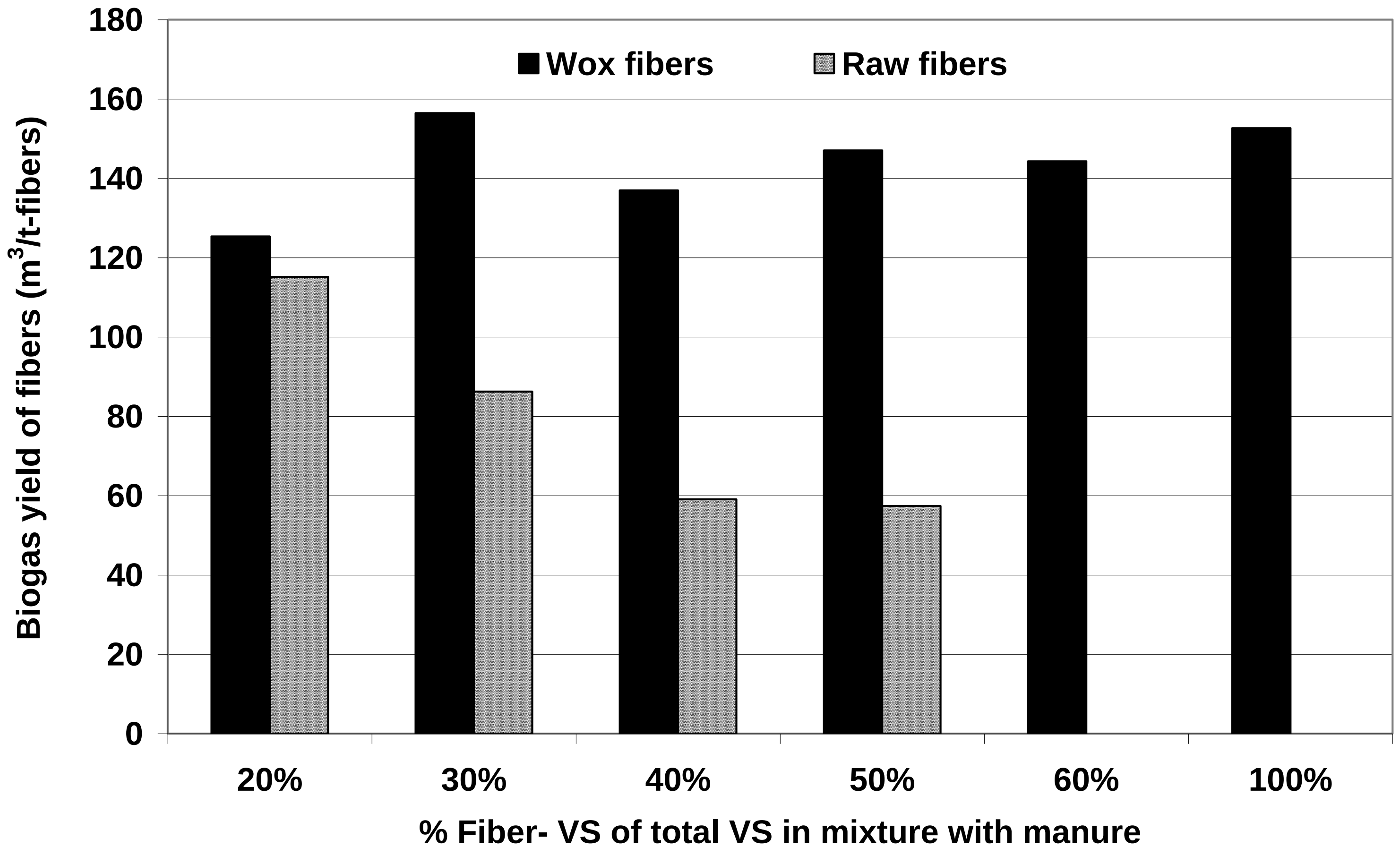
Batch experiments – wet oxidation

Wet oxidation of whole manure



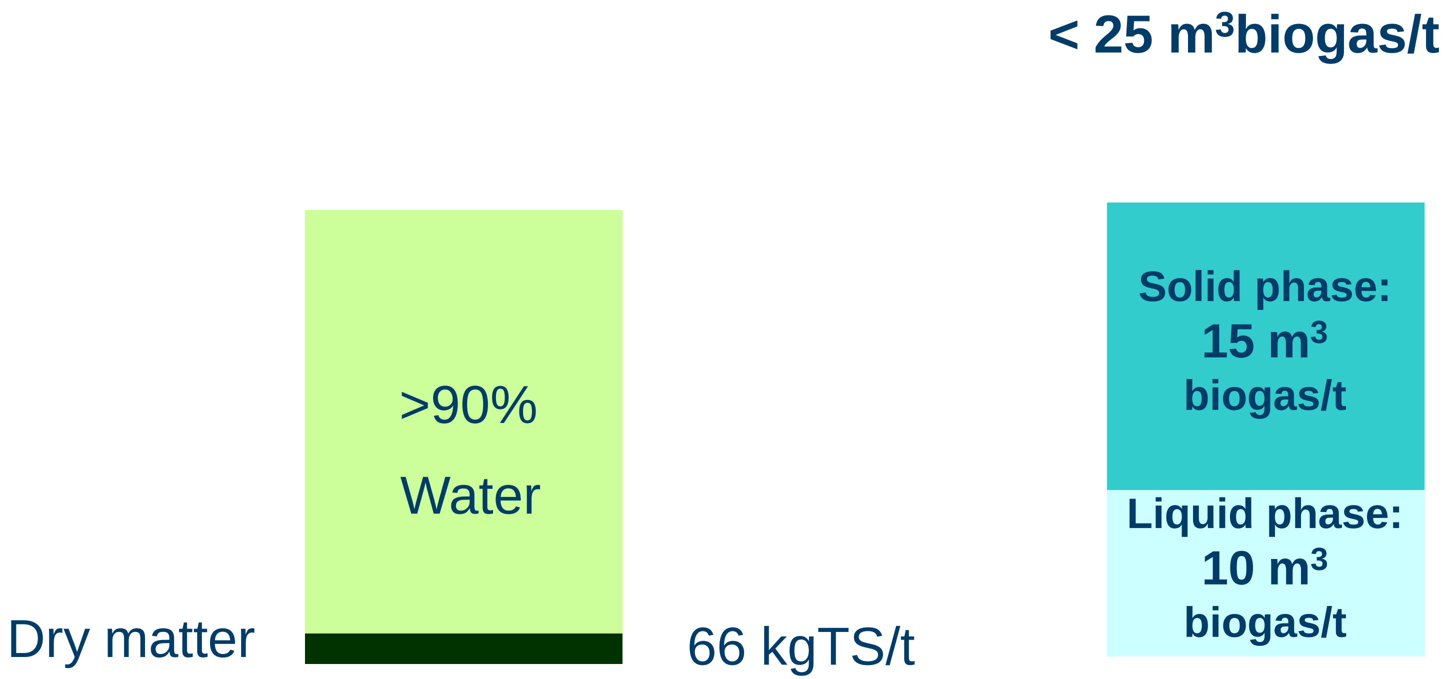



R1 biogas yield
R1 biogas yield avg
R2 biogas yield
R2 biogas yield avg
Wet oxidized manure feed to R1
Biogas yield (L/kg-VS)
700
600
500
400
300
200
100
0
0
10
20
30
40
50
60
70
Time (days)



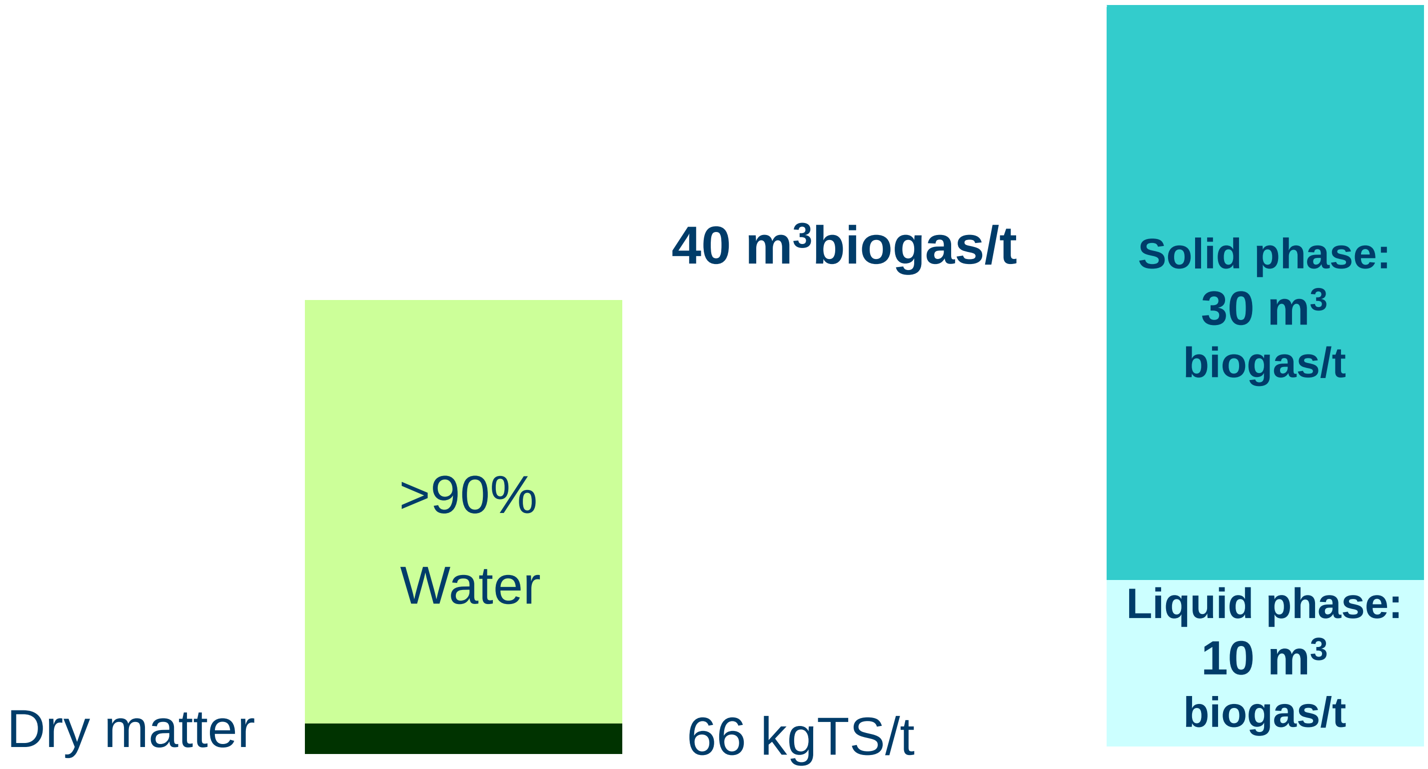
180
160
140
120
100
80
60
40
20
0
Wox fibers
Raw fibers
Biogas yield of fibers (m3/t-fibers)
20%
30%
40%
50%
60%
100%
% Fiber- VS of total VS in mixture with manure

Biogas yield from manure

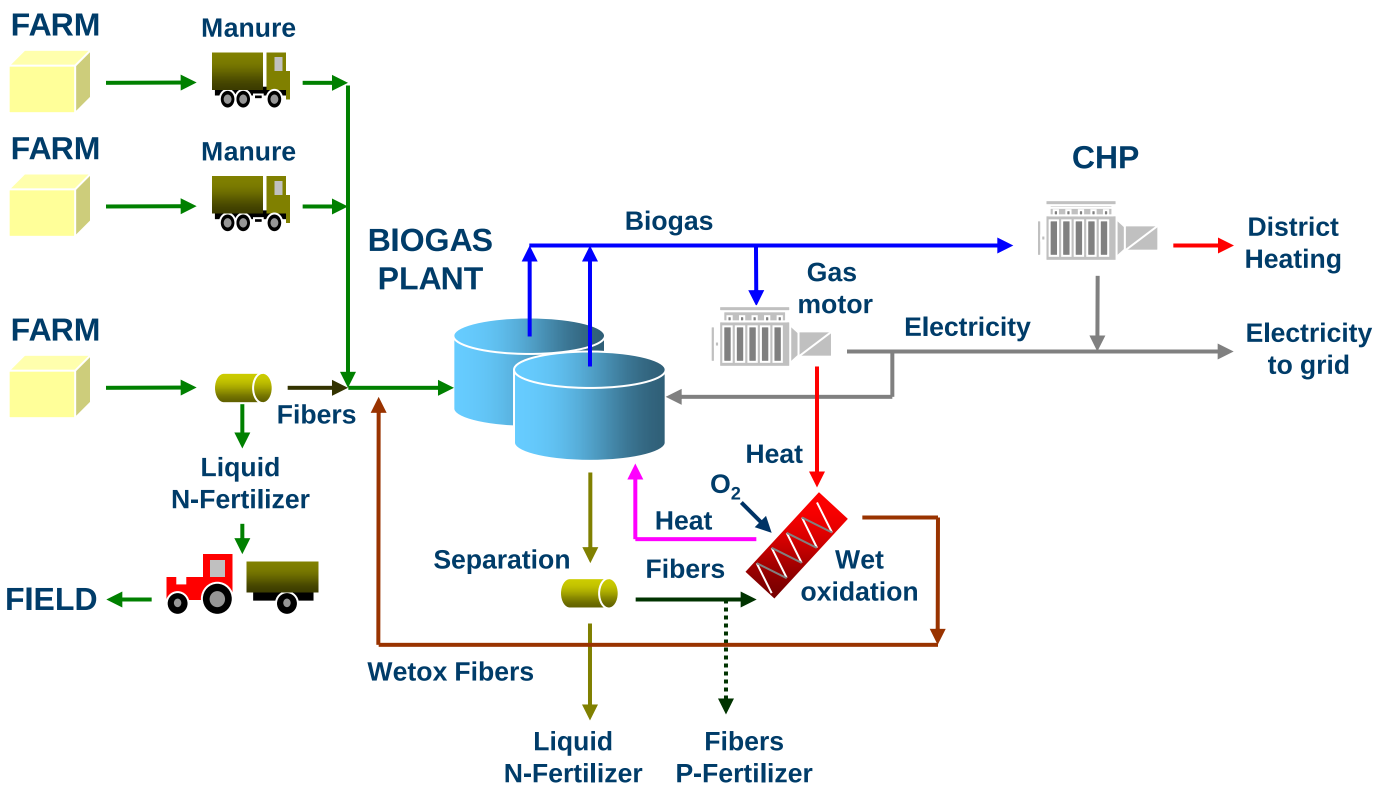


Increase of biogas yield per ton manure

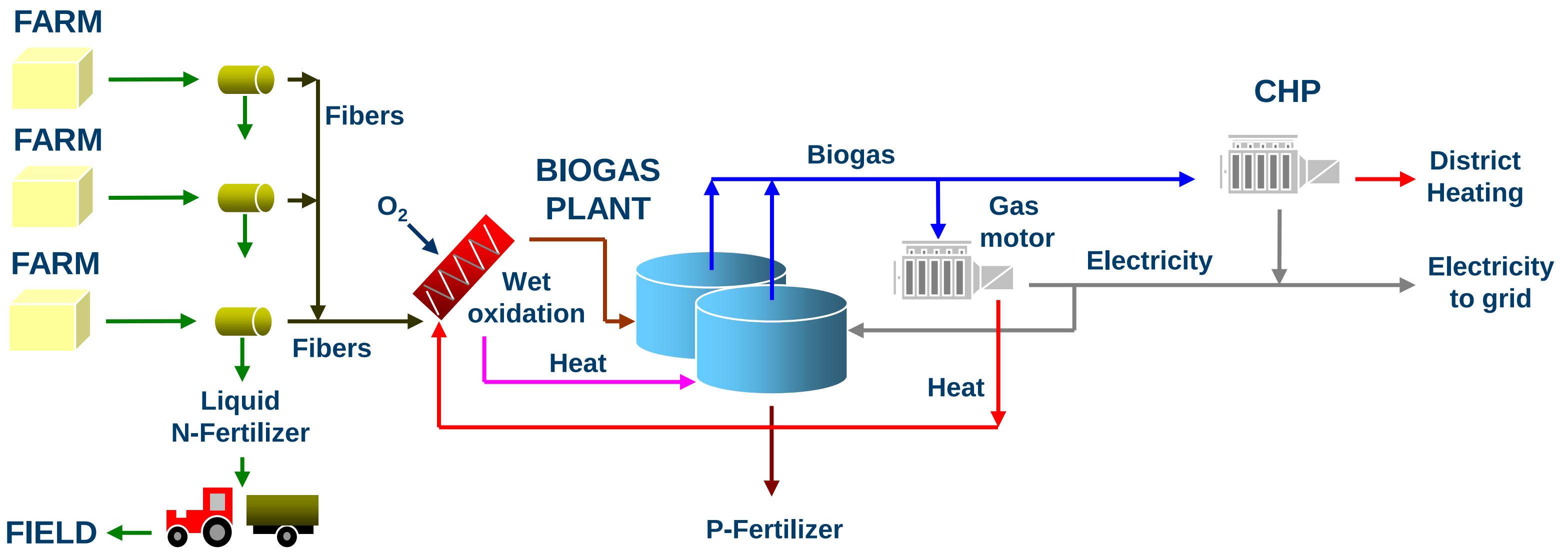
Wet oxidation of fibers:

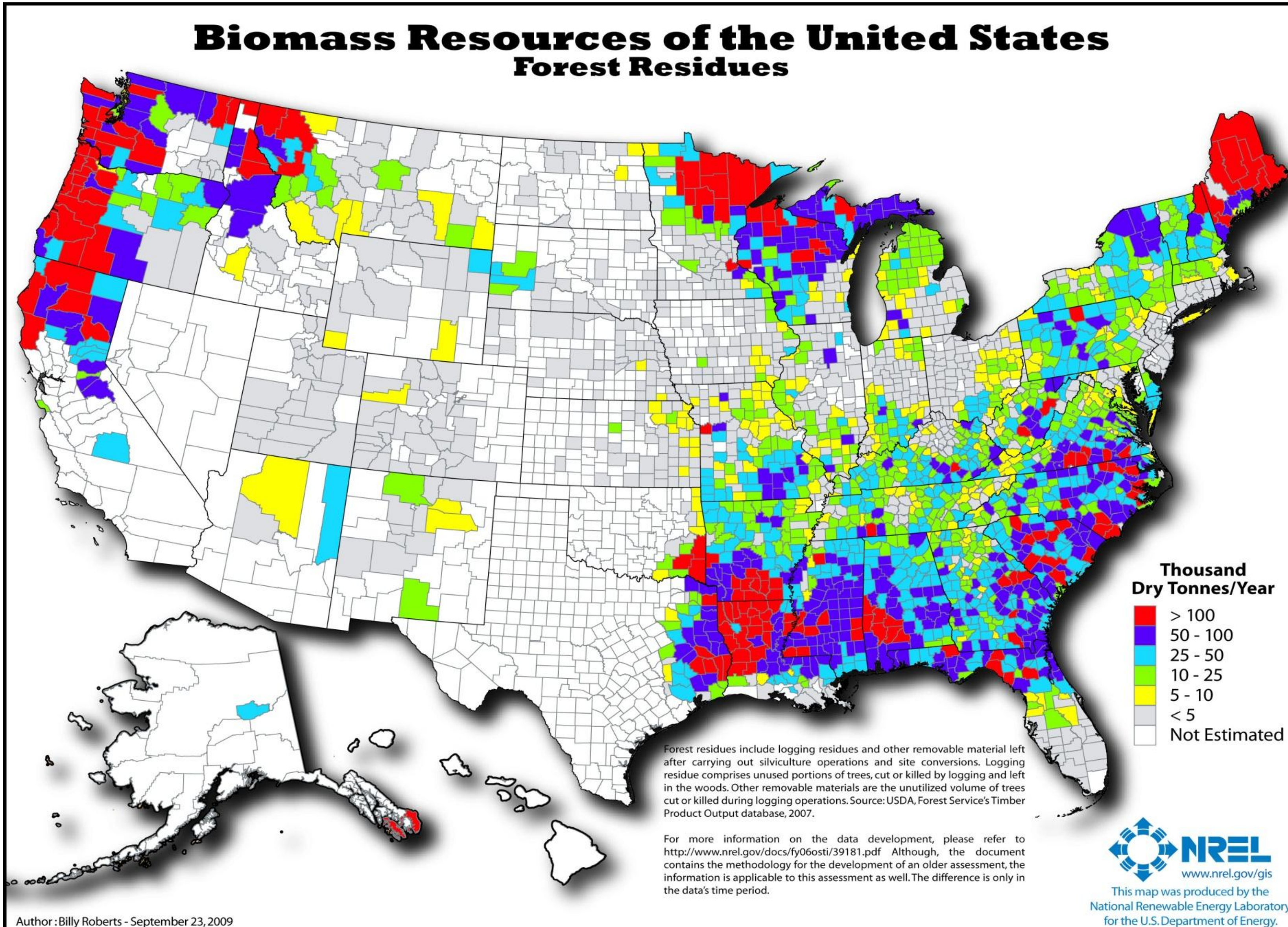


Separation of fibers + wet oxidation of recycled fibers

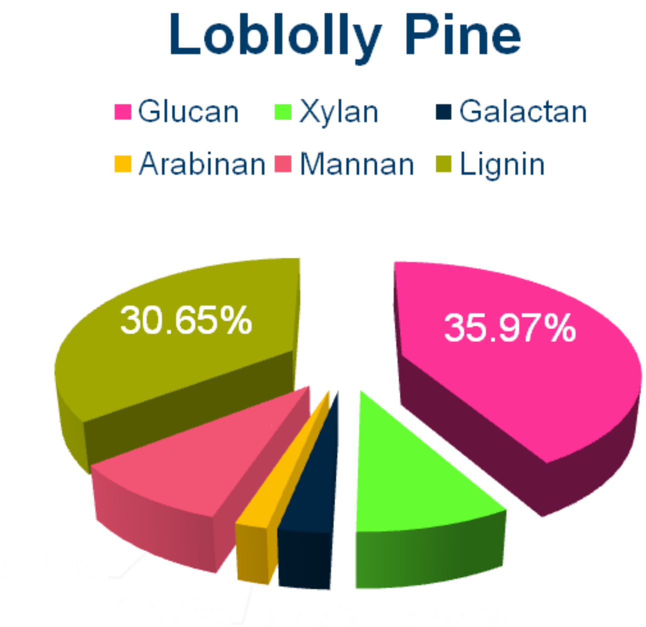


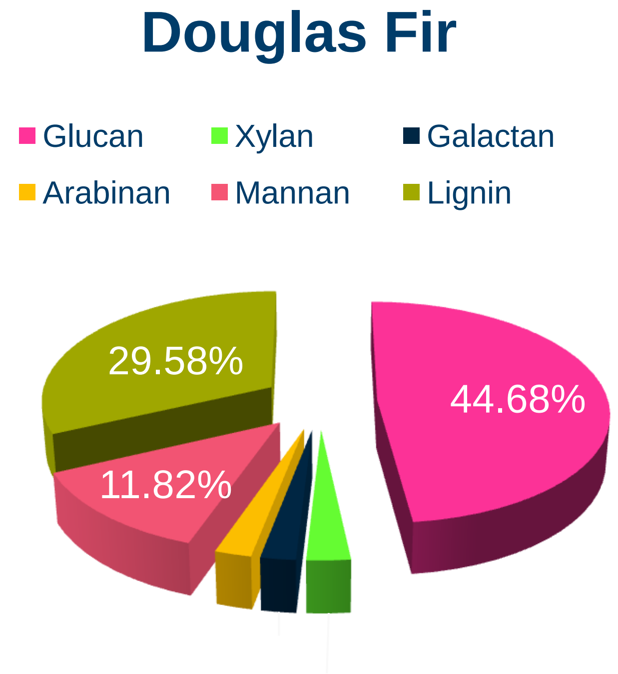
Separation + wet oxidation of fibers




Biomass Resources of the United States
Forest Residues
Thousand
Dry Tonnes/Year
> 100
50 - 100
25 - 50
10 - 25
5 - 10
< 5
Not Estimated
Forest residues include logging residues and other removable material left after carrying out silviculture operations and site conversions. Logging residue comprises unused portions of trees, cut or killed by logging and left in the woods. Other removable materials are the unutilized volume of trees cut or killed during logging operations. Source: USDA, Forest Service's Timber Product Output database, 2007.
For more information on the data development, please refer to http://www.nrel.gov/docs/fy06osti/39181.pdf Although, the document contains the methodology for the development of an older assessment, the information is applicable to this assessment as well. The difference is only in the data's time period.
NREL
www.nrel.gov/gis
This map was produced by the National Renewable Energy Laboratory for the U.S. Department of Energy.
Author : Billy Roberts - September 23, 2009

RAW COMPOSITION

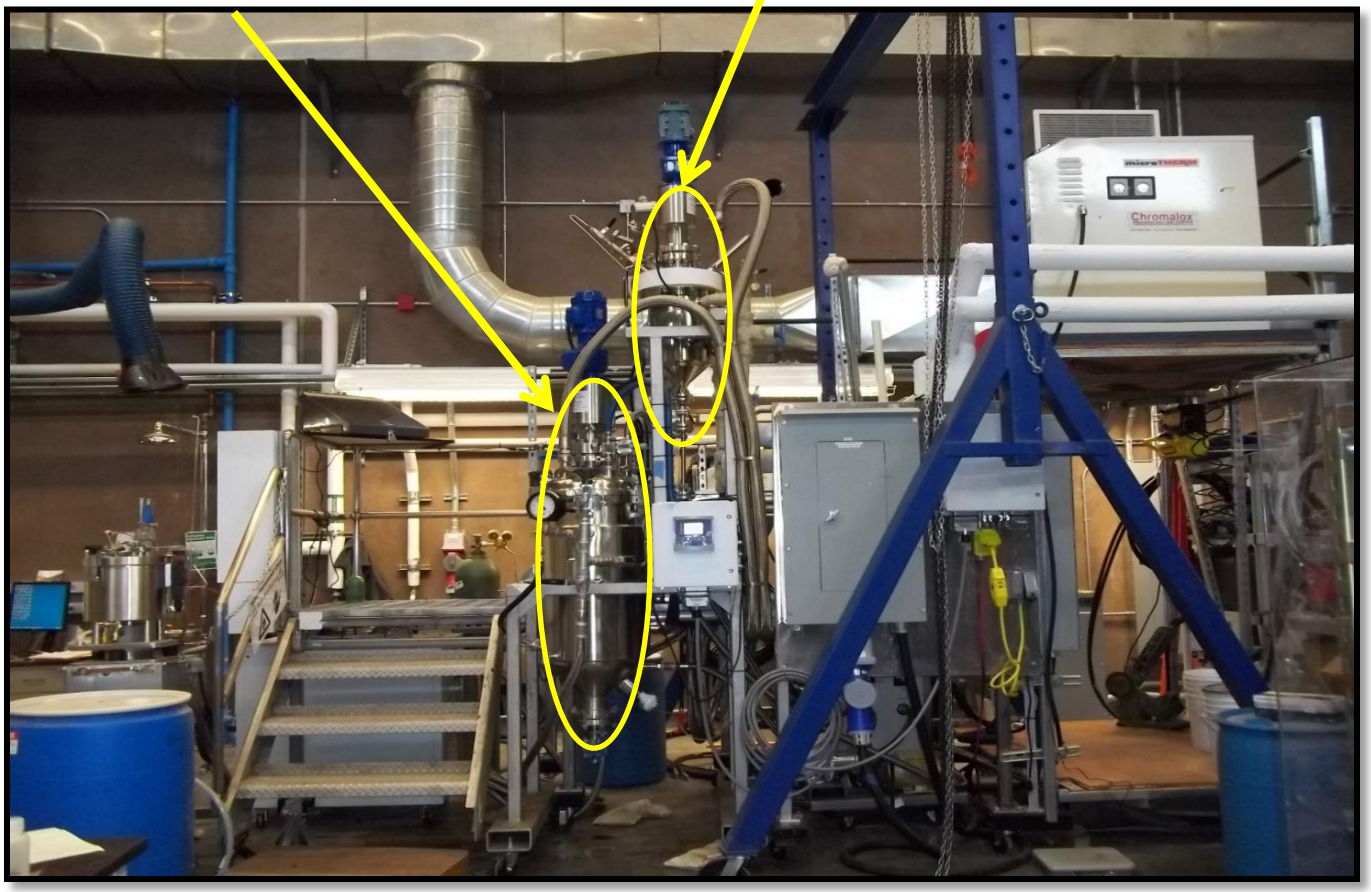




PILOT PLANT

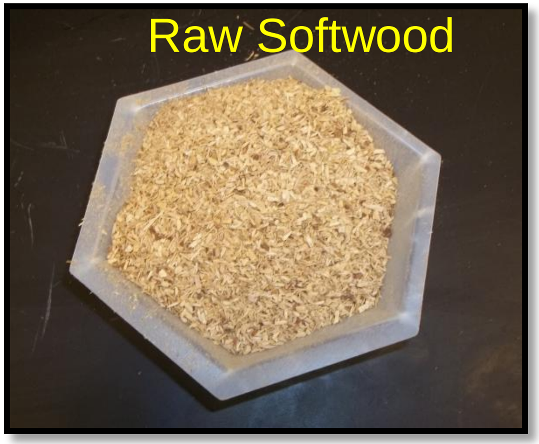
Flash Tank

Pretreatment Reactor



Softwood to hydrolysate and sugars

Milling



Pretreatment

Enzymatic

Hydrolysis

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WEx Pretreatment - Material Balance - Douglas Fir - FS-01

Stream 1 – Douglas Fir, Water & Oxygen

20% DM

100 kg of Slurry

Glucan	8.94	kg
Xylan	0.50	kg
Arabinan	0.42	kg
Galactan	0.40	kg
Mannan	2.36	kg
Lignin	5.92	kg

WEx Pretreatment
10 L Pilot Plant
(185 °C, 25 min, 5 bar Oxygen)

Stream 3 – Solids

Solids Yield	19.75	kg	Normalized data
Glucan	7.87	kg	88%
Xylan	0.10	kg	21%
Arabinan	0.15	kg	36%
Galactan	0.11	kg	28%
Mannan	1.33	kg	56%
Lignin	5.76	kg	97%

Stream 2 – Liquid

Liquid Yield	80.25	kg	Normalized data
Glucose	0.77	kg	8%
Xylose	0.42	kg	74%
Arabinose	0.30	kg	62%
Galactose	0.33	kg	74%
Mannose	1.20	kg	46%
Lignin	0.07	kg	1%
HMF	0.01	kg	-
Furfural	0.07	kg	-
Acetic Acid	0.14	kg	-

	Monomers	Oligomers	
Glucose	0.34	0.43	kg
Xylose	0.22	0.20	kg
Arabinose	0.07	0.23	kg
Galactose	0.18	0.15	kg
Mannose	0.46	0.74	kg

Slurry → **Enzymatic Hydrolysis** (Enzymes; 4M KOH)
250 mL Shaker
Ctec2: 40 mg protein/ g glucan
Htec2: 10% of Ctec2
(90 Hrs, pH 5.0, 50°C)

→ Separation

Stream 5 – Solids

Solids Yield	10.08	kg	Normalized data
Glucan	1.09	kg	12%
Xylan	0.02	kg	3%
Arabinan	0.00	kg	0%
Galactan	0.02	kg	5%
Mannan	0.10	kg	4%
Lignin	5.65	kg	95%

Stream 4 – Liquid

Liquid Yield	89.92	kg	Normalized data
Glucose	8.79	kg	89%
Xylose	0.54	kg	96%
Arabinose	0.48	kg	100%
Galactose	0.43	kg	96%
Mannose	2.46	kg	94%
Lignin	0.17	kg	3%
HMF	0.04	kg	-
Furfural	0.27	kg	-
Acetic Acid	0.38	kg	-

	Monomers	Oligomers	
Glucose	8.60	0.19	kg
Xylose	0.53	0.01	kg
Arabinose	0.43	0.05	kg
Galactose	0.42	0.01	kg
Mannose	2.04	0.42	kg

Conversion

	Overall
Glucose	88.5%
Xylose	95.9%
Arabinose	99.8%
Galactose	96.0%
Mannose	93.6%
Lignin	2.9%

Recovery

	Overall
Glucose	101%
Xylose	99%
Arabinose	100%
Galactose	101%
Mannose	98%
Lignin	98%

Sugars Yields from Softwood

Type of Biomass	Type of Pretreatment	Pretreatment Temperature (^{0}C)-Time (min)	Enzymatic Hydrolysis	Theoretical Yield (Total Sugars)	Reference
Softwood	Two- step Steam Pretreatment	Stage 1: 190-2, 3% SO_2 Stage 2: 220-5, 3% SO_2	2% DM	80%	Söderström J. et al. (2002)
Pinus rigida	Organosolv	210-10, 1% $MgCl_2$	1% DM	75.88%	Park N. et al. (2010)
Bettle Killed Lodgepole	One step Steam Pretreatment	200-5, 4% SO_2	2% DM	75%	Ewanick S. et al. (2007)
Loblolly pine	Wet Explosion	180-20, 6 bar O_2	25% DM	**96.00%**	Rana D. et al. (2012)

Material Balance - Douglas Fir - FS-03

Feed (1): Douglas Fir, Water & Oxygen

20% DM

100 kg of Slurry

Glucan	8.30	kg
Xylan	1.87	kg
Arabinan	0.66	kg
Galactan	0.62	kg
Mannan	1.50	kg
Lignin	6.78	kg

WEx Pretreatment
10 L Pilot Plant
(185 °C, 25 min, 5 bar Oxygen)

Slurry →

Enzymatic Hydrolysis
250 mL Shaker @ 13% DM
Ctec2: 40 mg protein/ g glucan
Htec2: 10% of Ctec2
(90 Hrs, pH 5.0, 50°C)

4M KOH

→ Separation

(3) Solids

Solids Yield	17.00	kg	Normalized data
Glucan	7.52	kg	*91%*
Xylan	0.97	kg	*52%*
Arabinan	0.53	kg	*81%*
Galactan	0.49	kg	*79%*
Mannan	0.49	kg	*33%*
Lignin	6.44	kg	*95%*

(2) Liquid

Liquid Yield	83.00	kg	Normalized data
Glucose	1.19	kg	*13%*
Xylose	0.25	kg	*12%*
Arabinose	0.12	kg	*15%*
Galactose	0.30	kg	*44%*
Mannose	0.54	kg	*32%*
Lignin	0.44	kg	*6%*
HMF	0.05	kg	-
Furfural	0.16	kg	-
Acetic Acid	0.44	kg	-

	Monomers	Oligomers	
Glucose	0.56	0.64	kg
Xylose	0.17	0.08	kg
Arabinose	0.07	0.05	kg
Galactose	0.22	0.09	kg
Mannose	0.43	0.12	kg

(5) Solids

Solids Yield	7.00	kg	Normalized data
Glucan	2.86	kg	*34%*
Xylan	0.25	kg	*13%*
Arabinan	0.06	kg	*9%*
Galactan	0.06	kg	*10%*
Mannan	0.29	kg	*19%*
Lignin	4.48	kg	*66%*

(4) Liquid

Liquid Yield	93.00	kg	Normalized data
Glucose	3.79	kg	*41%*
Xylose	1.31	kg	*61%*
Arabinose	0.46	kg	*62%*
Galactose	0.43	kg	*63%*
Mannose	0.94	kg	*56%*
Lignin	0.35	kg	*5%*
HMF	0.06	kg	-
Furfural	0.07	kg	-
Acetic Acid	0.33	kg	-

	Monomers	Oligomers	
Glucose	3.72	0.07	kg
Xylose	0.69	0.62	kg
Arabinose	0.00	0.46	kg
Galactose	0.19	0.24	kg
Mannose	0.36	0.58	kg

Conversion

	Overall
Glucose	54.8%
Xylose	81.7%
Arabinose	82.2%
Galactose	84.0%
Mannose	74.8%
Lignin	7.0%

Recovery

	Overall
Glucose	101%
Xylose	99%
Arabinose	94%
Galactose	98%
Mannose	100%
Lignin	95%

Conclusion

- Wet explosion was found to be well suited as a pretreatment method for production of ethanol and biogas from agricultural residues
- Wet explosion was further found to produce high sugar yields (both C6 and C5) from softwood
- Enzyme cost demands that hydrolysis of pretreated materials has to be optimized
- Economics might be more favorable for non-maximum sugar production from biomass materials
- Pretreatment of forest slash needs upfront processing before pretreatment

Thank you and Questions

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