Process Scale-up of MSW/CS Blends Conversion into Sugars

LBNL- ABPDU
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Background:
IL Acidolysis for Biomass Deconstruction

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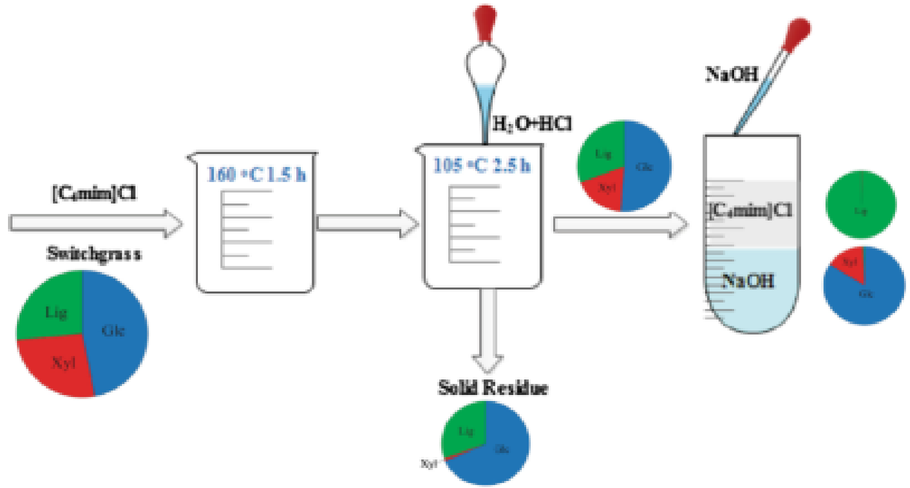
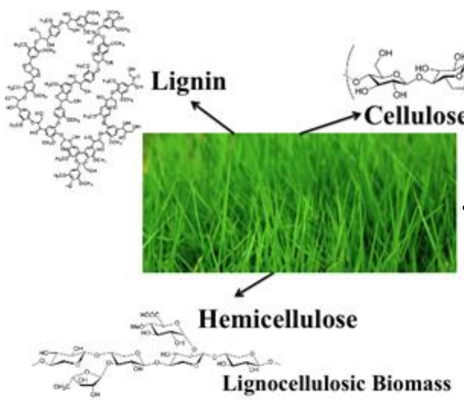
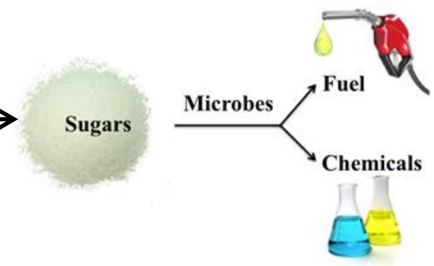




IL Pretreatment → No Solid/Liquid Separation → Acidolysis



One-Pot IL Process –
Enzyme Free and Wash Free

Sugar Extraction, IL and Lignin Recovery

Universal Feedstock Utilization

Sun et al., 2013, Biotechnology for Biofuels, 6:39

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Low Cost Feedstocks - MSW

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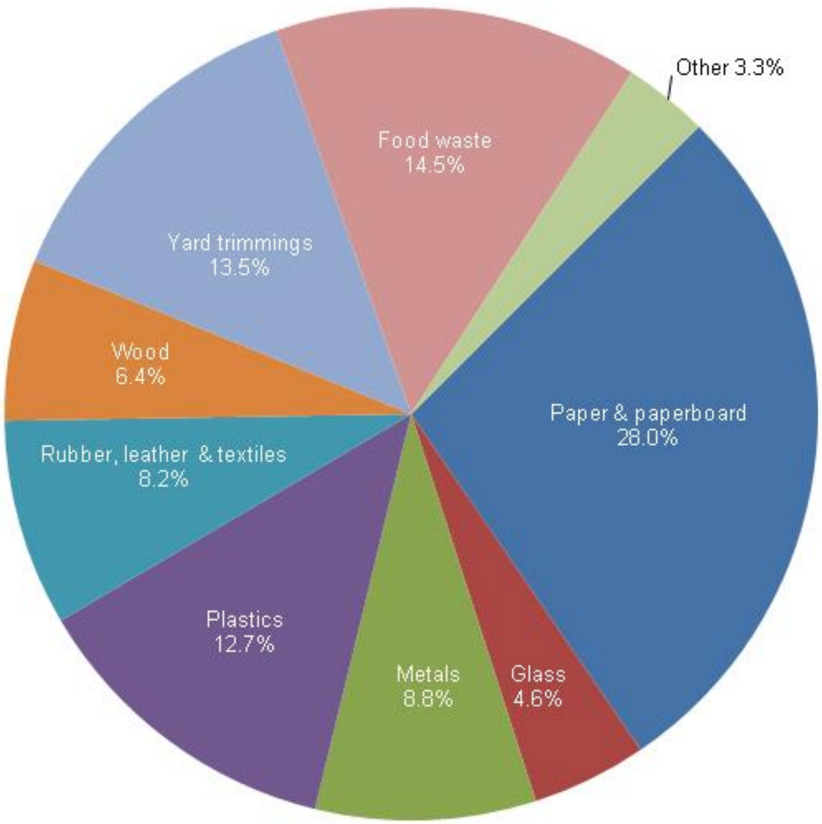
- **Advantages**
  - Year-round availability
  - Low or negative cost
  - Collection infrastructure
  - Abundance and renewable

- **Disadvantages**
  - Highly variable
    - Season
    - Year
    - Region
  - Low quality
    - Sorting
    - Upgrading

Figure 4. Total MSW Generation (by material), 2011
250 Million Tons (before recycling)



Source: http://www.epa.gov/epawaste/nonhaz/municipal/index.htm

MSW/CS Blends Compositions FY14

CS/MSW	Ash (%)	Glucan (%)	Xylan (%)	Glucan+Xylan (%)
100:0	3.0	33.2	20.8	50.8
90:10	3.8	35.5	19.7	55.2
80:20	4.6	37.7	18.6	56.3
70:30	5.4	40.0	17.6	57.6
60:40	6.2	42.2	16.5	58.7
50:50	7.0	44.5	15.4	59.9
0:100	10.9	50.8	10.0	60.8

MSW: Paper mix waste materials containing glossy paper, non-glossy paper, glossy cardborad, non-glossy cardboard

- MSW/CS blends have the great potential to meet quality requirements.

Sun et al., 2015, Bioresour Technol, 186: 200-206

Least Cost Formulation Output for Midwest Corn Stover and MSW Blend

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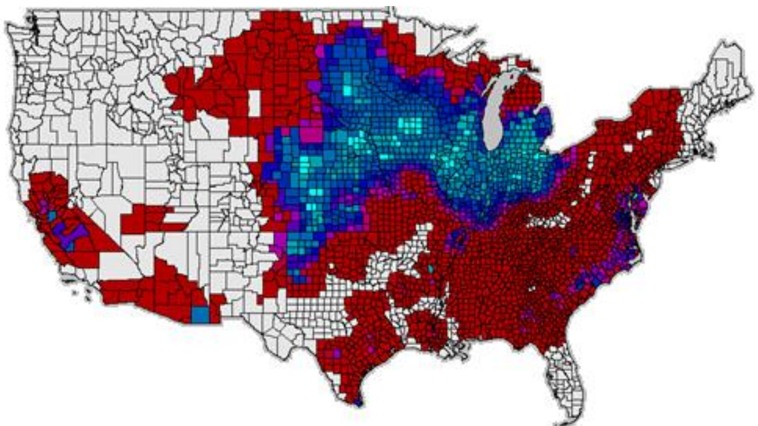
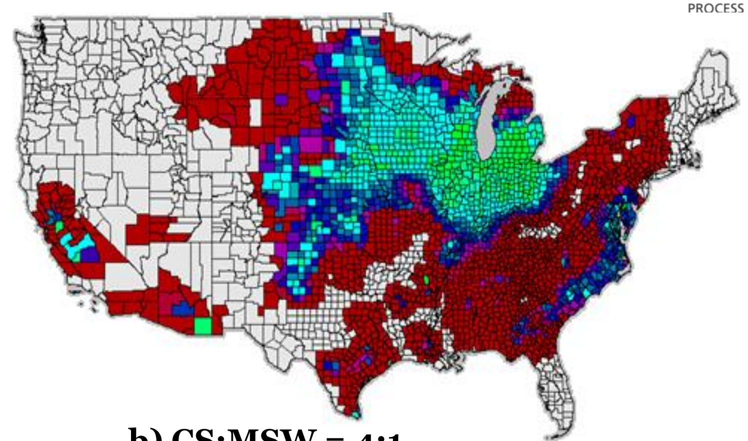
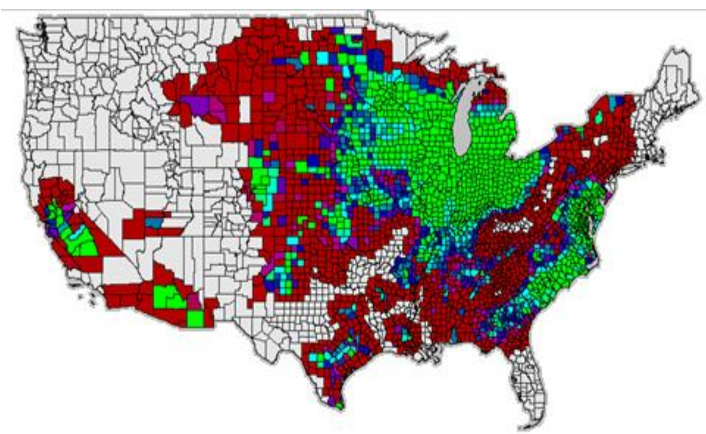


a) Corn Stover



b) CS:MSW = 4:1



c) CS:MSW = 1:1

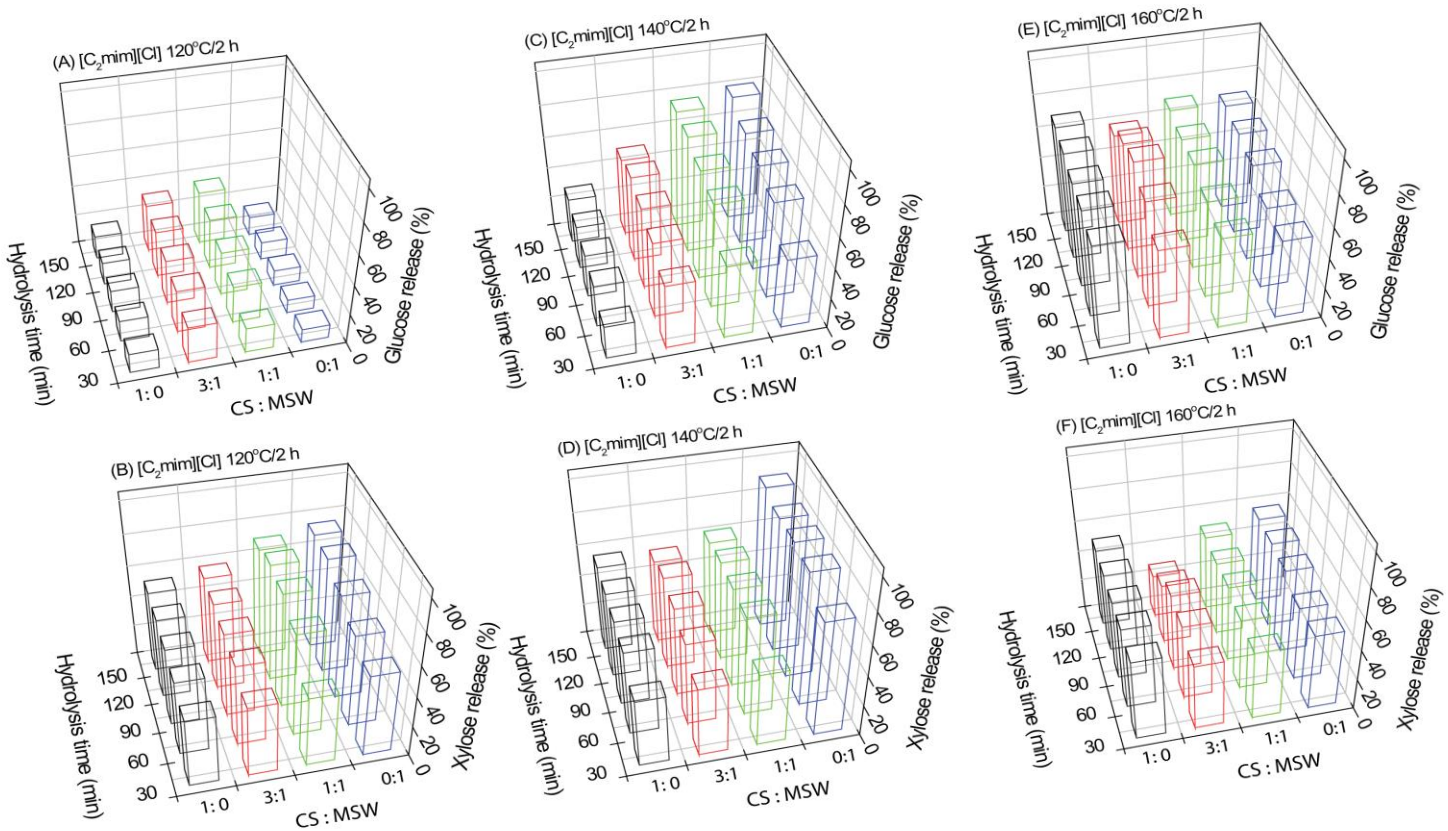
70$/ton ton | 80$/ton | >100$/

DOE target

Sun et al., 2015, Bioresour Technol, 186: 200-206

- MSW/CS blends have the great potential to meet the cost target.

IL Acidolysis of MSW/CS blends at 30 mL



- **The highest glucose (80.6%) and xylose (90.8%) yields are obtained after pretreatment of MSW at 140 ºC for 2 h.**
- **MSW/CS blends generate higher sugar yields than CS at milder conditions.**
- **Similar results were found for both 1-ethyl-3-methylimidazolium chloride and1-butyl-3-methylimidazolium chloride**

Sun et al., 2015, Bioresour Technol, 186: 200-206

MSW Biomass Blends Conversion Scale-up at ABPDU

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3 x 10L Parr Reactors and 210L Andritz Reactor

4 x 2L and 1 x 50L IKA Reactor

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Background:
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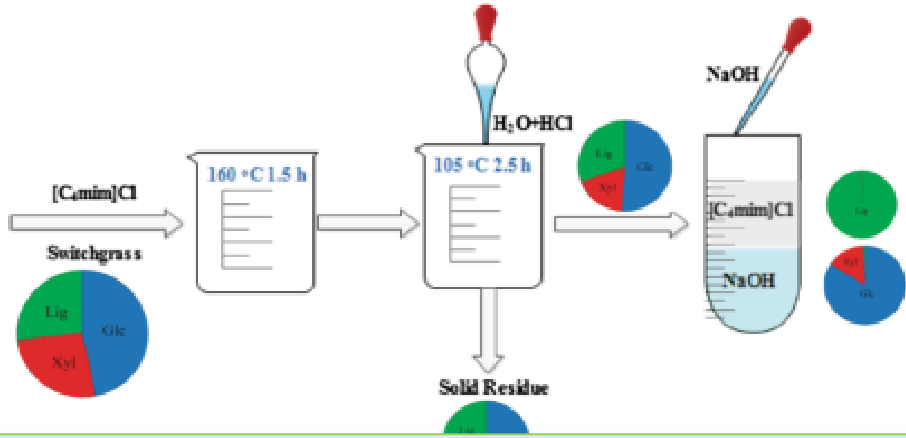

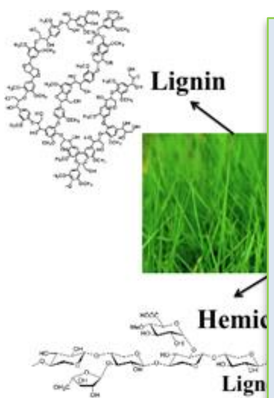

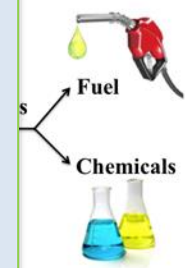

Process Scale-up Challenges:

- Reactor material compatibility with IL and acid
- Mixing at high solid loadings
- Acid/water injection at high temperature
- Safety of reactor operation and material handling

Sun et al., 2013, Biotechnology for Biofuels, 6:39

Coupon Testing for Material Compatibility

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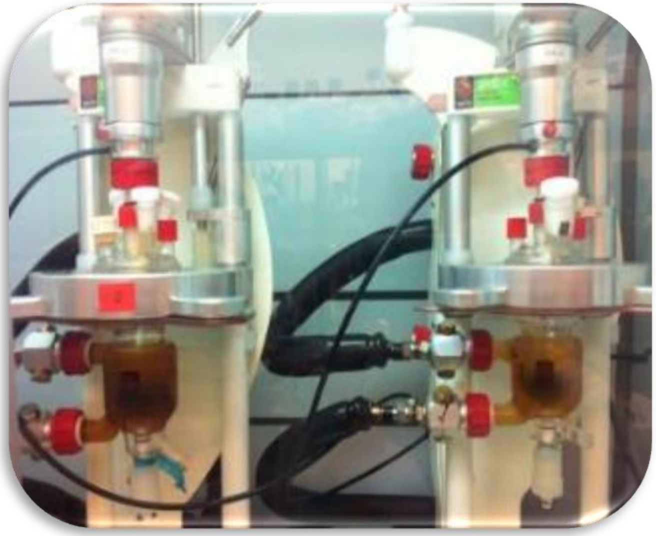
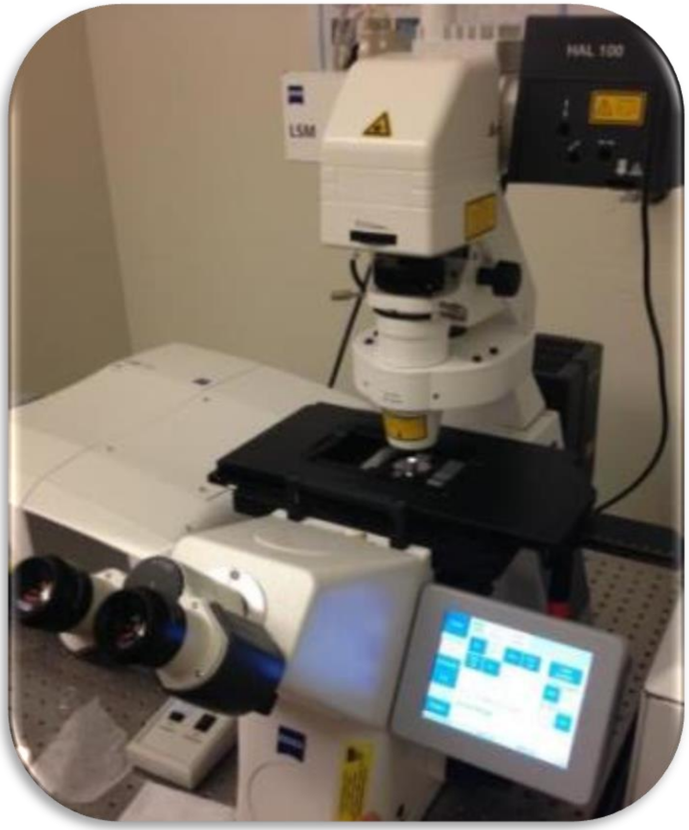


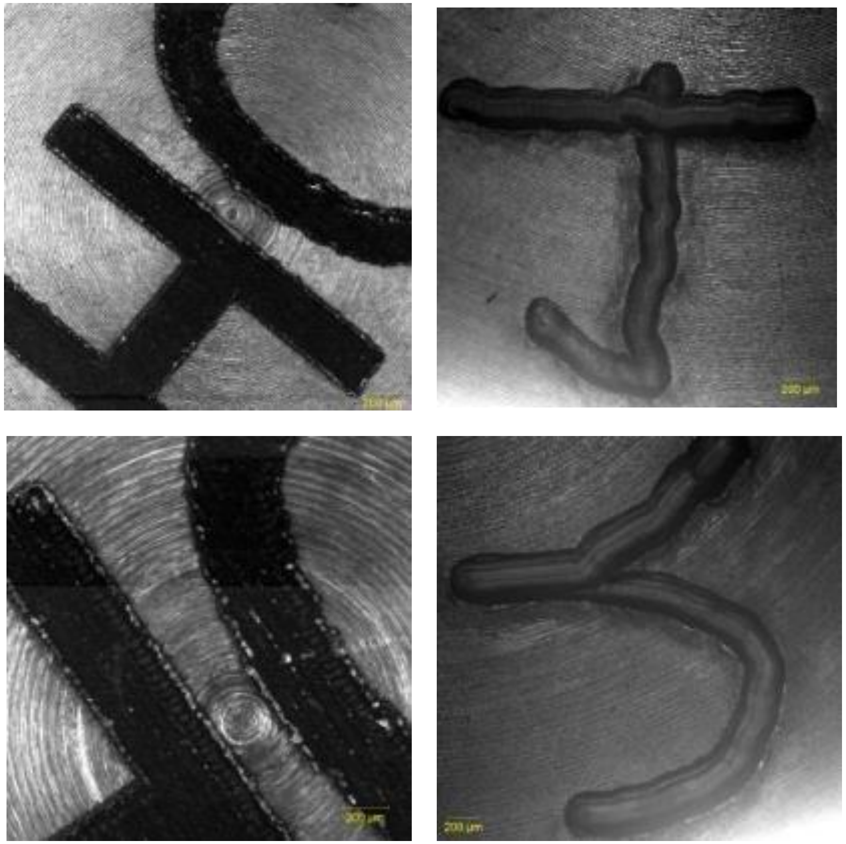
Hastelloy C276 Coupons, IL Pretreatment @160ºC & 3h, Acidolysis @ 105ºC & 3 h, Incubation @ 25ºC overnight

1-ethyl-3-methylimidazolium chloride
1-butyl-3-methylimidazolium chloride

6 cycles, 24 hours / cycle

Imaging – Surface Changes





Very minor surface corrosion was observed.

Modification of Impeller for High Solids

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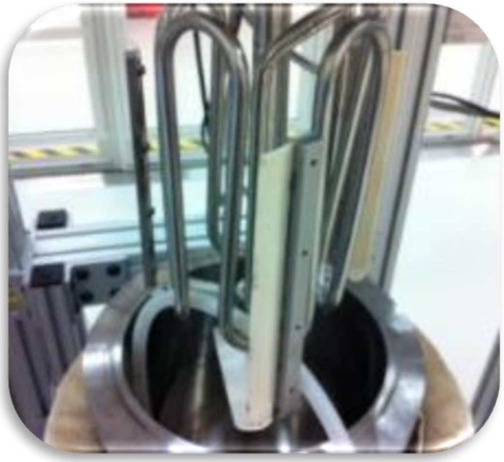
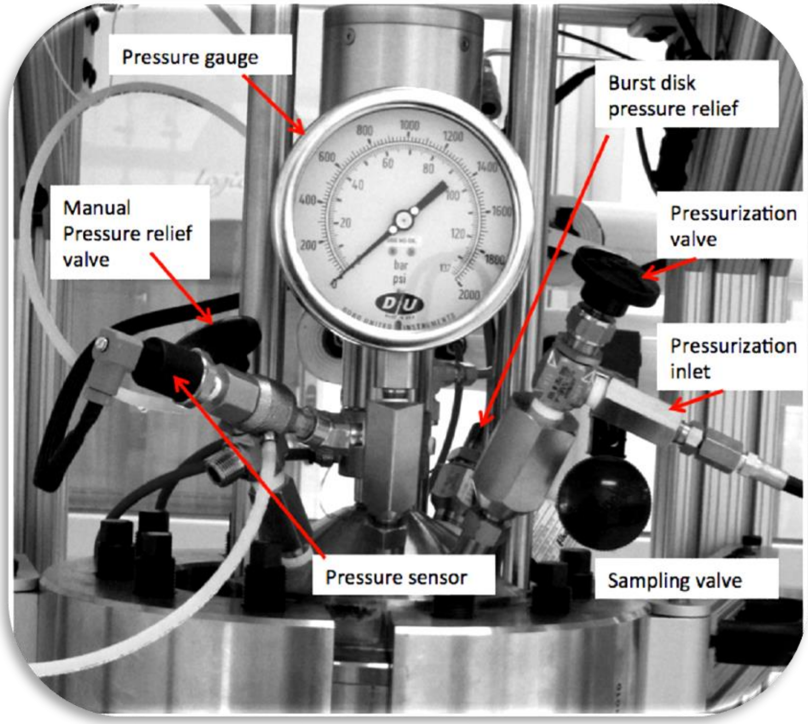
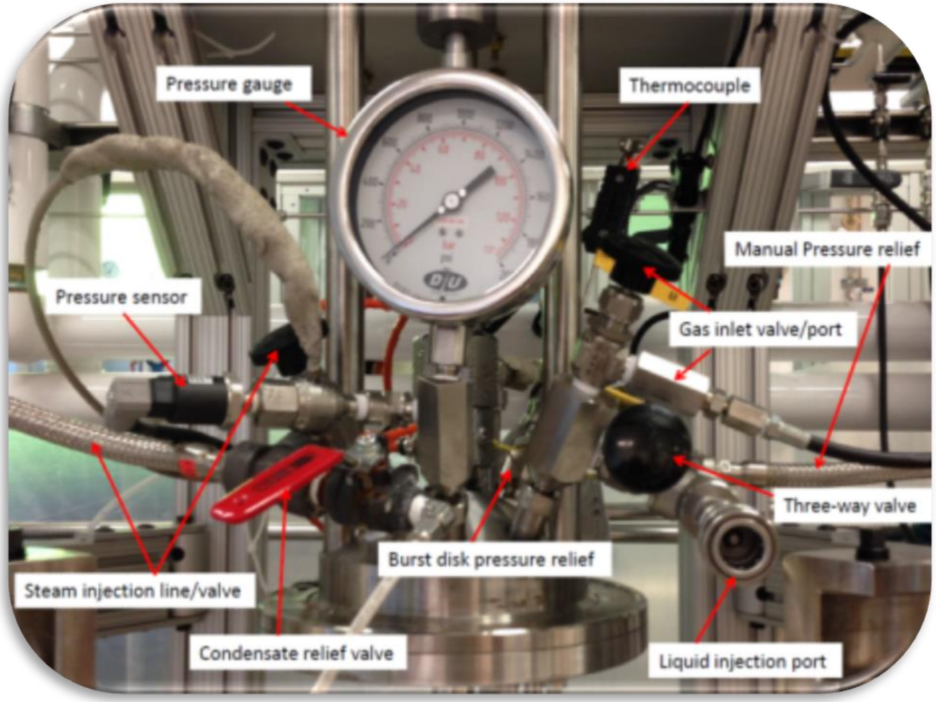


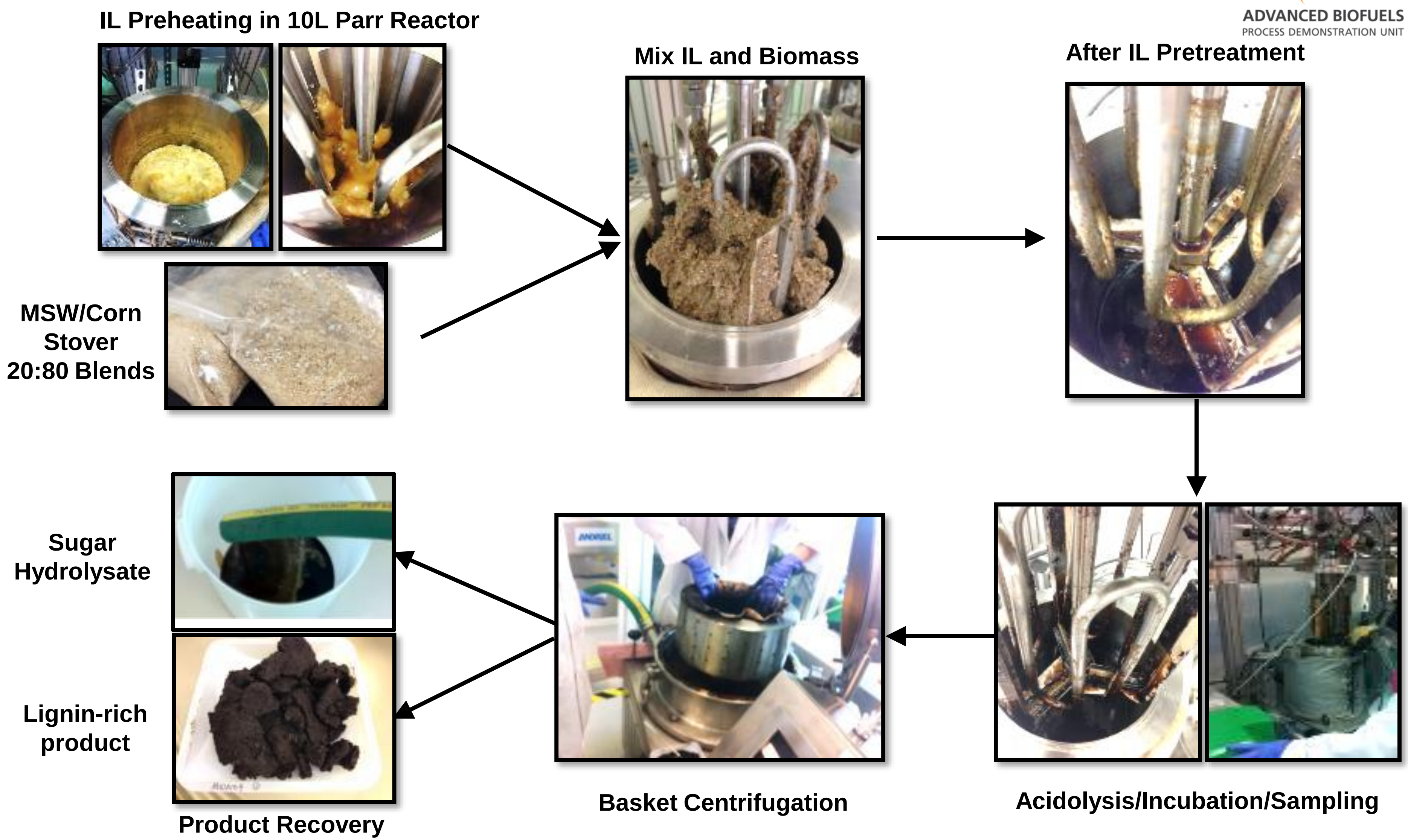
Magnetic drive **Self-sealing packed gland drive** **Anchor impeller**

Customization for Acid/Water Injection

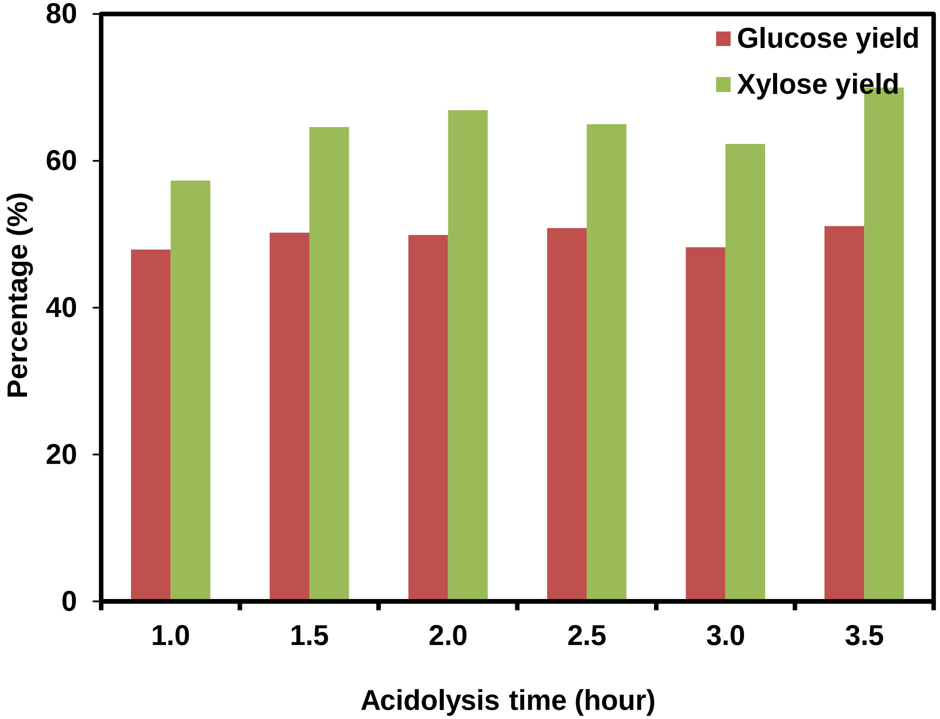


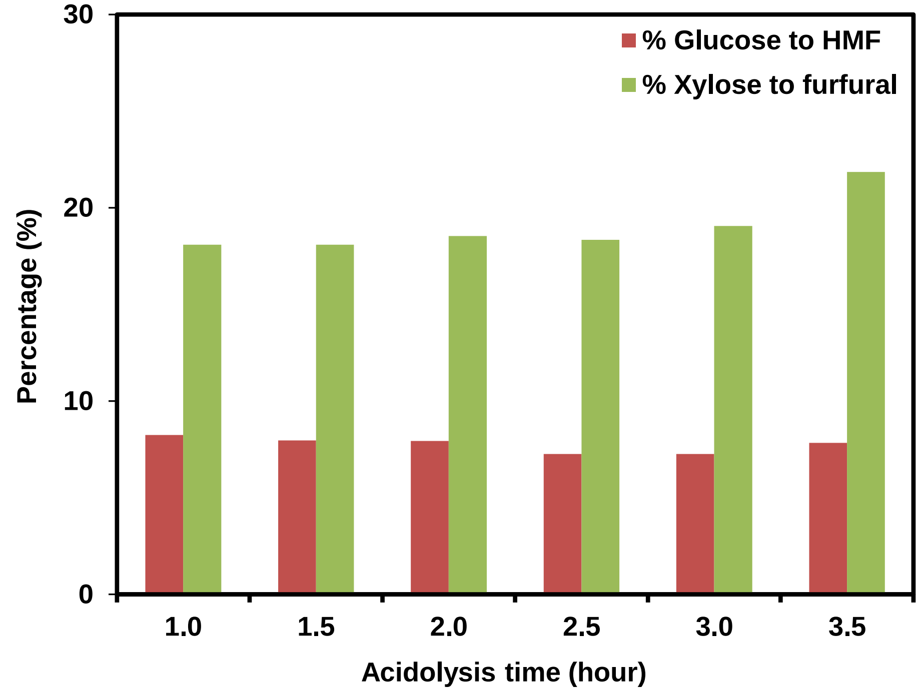


IL Acidolysis Scale-up Process Flow



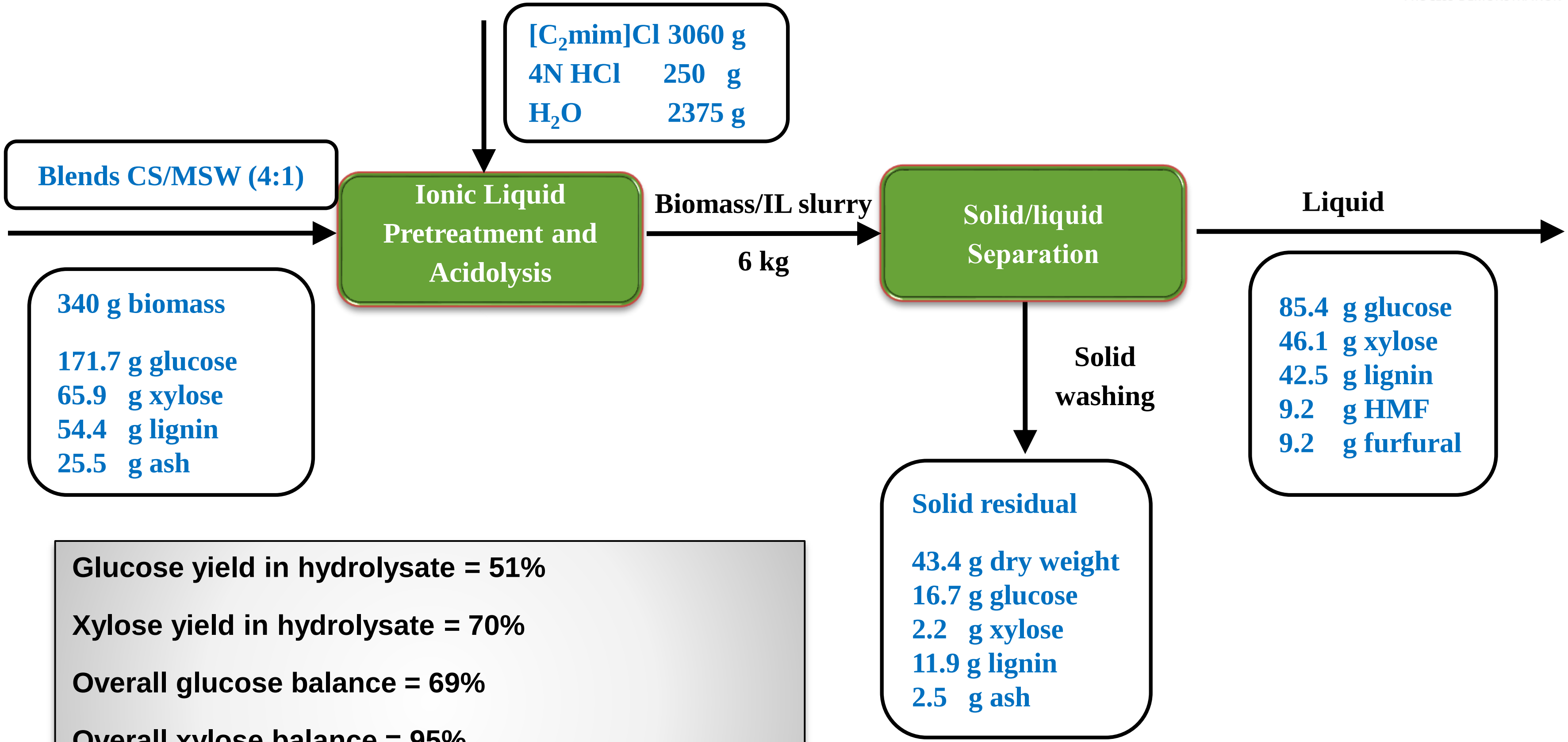
Acidolysis with $[C_2mim]Cl$ Pretreatment at 6L





- High xylose (70%) and glucose (51%) release was obtained for $[C_2mim]Cl$ pretreatment @140 ºC, 2h, 10% TS.
- Up to 8% of glucose was converted to HMF and 22% of xylose was converted to furfural after acidolysis.

Mass Balance



Glucose yield in hydrolysate = 51%

Xylose yield in hydrolysate = 70%

Overall glucose balance = 69%

Overall xylose balance = 95%

Overall lignin recovery from solid stream = 22%

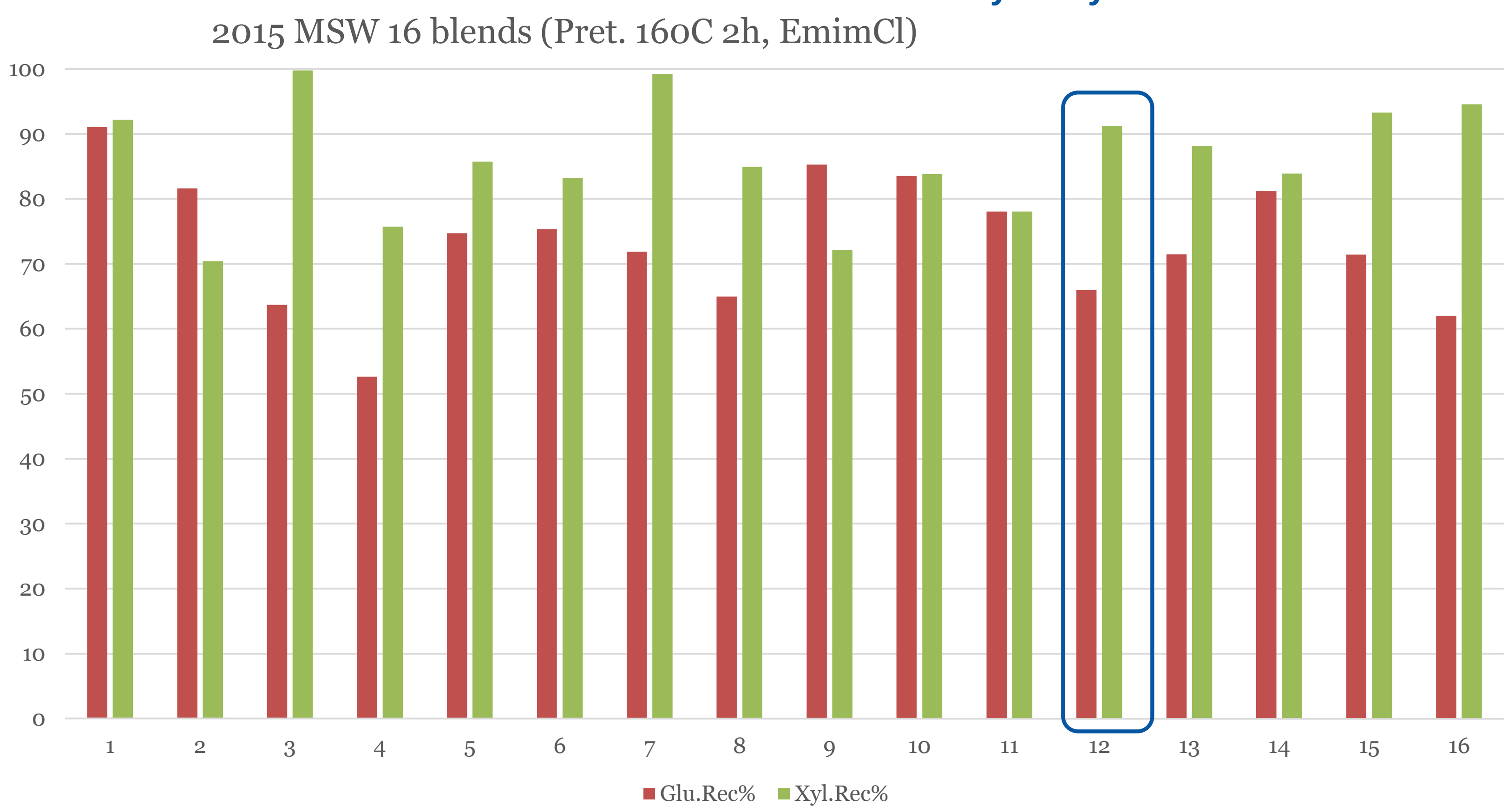
Blends Screening FY15

New blends 2015 (%)					
No.	Corn stover	Switchgrass	Grass clippings	MSW	Abbr.
1	90		10		CG9:1
2	80		20		CG8:2
3	70		30		CG7:3
4	60		40		CG6:4
5		90	10		SG9:1
6		80	20		SG8:2
7		70	30		SG7:3
8		60	40		SG6:4
9	90	10			CS9:1
10	80	20			CS8:2
11	90			10	CM9:1
12	80			20	CM8:2
13	70			30	CM7:3
14		90		10	SM9:1
15		80		20	SM8:2
16		70		30	SM7:3

MSW: The non-recyclable paper consisted of aseptic and polycoat containers and packaging, food soiled paper, shredded paper and waxed or coated papers and cardboard.

Small Scale Results Using Tube Reactor

65% glucose, 91 % xylose yield



Sugar Yield Summary

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Run	Biomass	Ionic liquids	Solid Loadings	T (°C)/t (h)	Glucose Yield (%)	Xylose Yield (%)	Solid Recovery (%)
1	A	$[C_4C_1mim]Cl$	10	140/2	56.2	84.3	13.2
2		$[C_4C_1mim]Cl$	10	160/2	29.0	50.0	4.5
3		$[C_2C_1mim]Cl$	10	140/2	49.7	70.0	12.8
4		$[C_2C_1mim]Cl$	15	140/2	38.2	55.4	34.8
5		$[C_4C_1mim]Cl$	15	160/2	46.1	52.1	N/A
6		$[C_2C_1mim]Cl$	15	160/2	32.8	40.7	N/A
7		$[C_4C_1mim]Cl$	10	140/2	36.7	76.0	N/A
8	B	$[C_4C_1mim]Cl$	10	140/2	38.2	50.7	16.9
9		$[C_4C_1mim]Cl$	10	150/2	58.9	38.2	5.9
10		$[C_4C_1mim]Cl$	10	160/2	70.9	53.1	0.4
11		$[C_4C_1mim]Cl$	15	160/2	63.3	41.0	2.8
12		$[C_4C_1mim]Cl$	10	120/2	53.7	51.0	13.6
13		$[C_2C_1mim]Cl$	10	120/2	44.5	46.9	21.3
14		$[C_2C_1mim]Cl$	10	140/2	53.6	35.2	11.7
15		$[C_2C_1mim]Cl$	10	160/2	NA	NA	6.8

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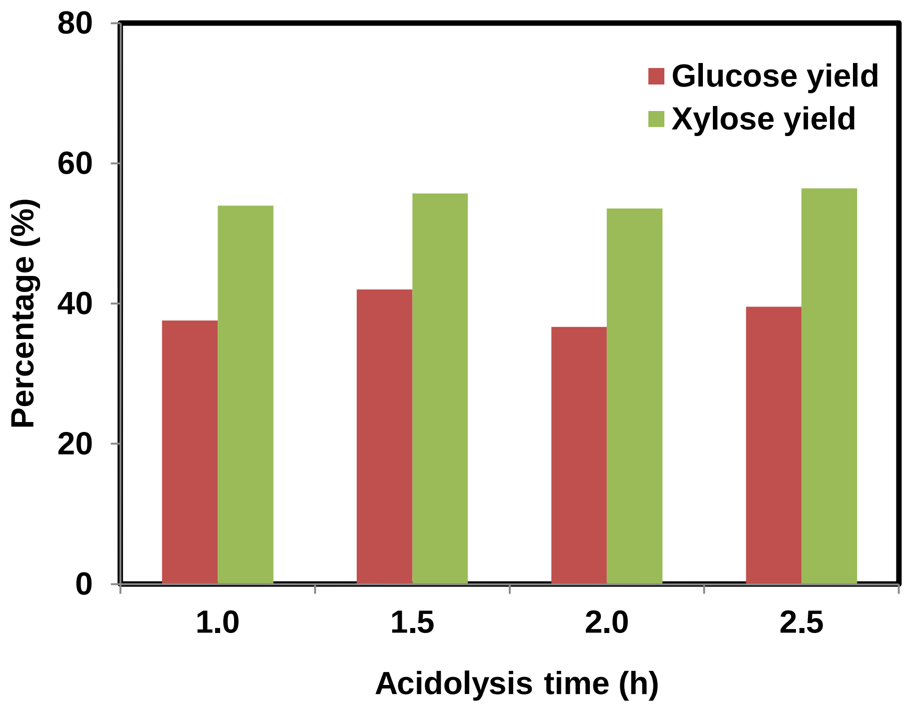
Composition of the Raw and Recovered Solids

Sample	Glucan (%)	Xylan (%)	Klason Lignin (%)	Acid soluble lignin (%)	Ash (%)	Total (%)
Raw MSW	43.85	24.16	17.11	0.32	3.42	88.87
MSW 08	44.20	2.33	41.94	0.32	8.71	97.50
MSW 09	34.94	1.30	37.72	0.66	22.74	97.35
MSW 10	4.84	0.71	49.06	0.45	42.39	97.44
MSW 11	4.81	0.00	38.39	0.33	41.75	85.29
MSW 12	59.00	0.91	19.77	0.53	13.83	94.05
MSW13	38.31	0.00	45.39	0.82	8.82	93.34
MSW14	14.86	0.00	66.41	0.00	11.44	92.70
MSW 15	34.72	17.60	22.14	1.77	2.04	80.86

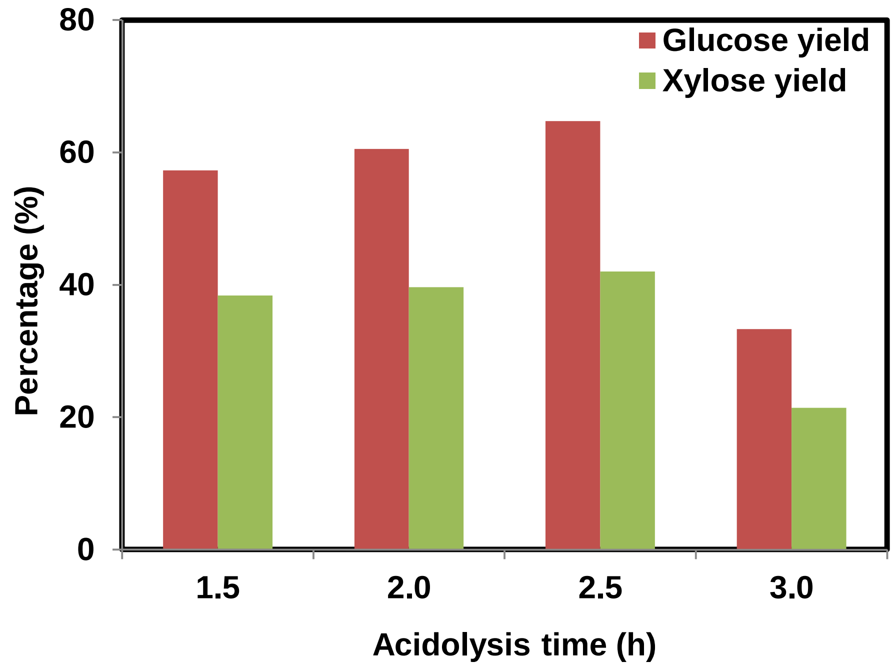
- **Recovered solids are rich in lignin and ash**

Sugar Yields with Different Pretreatment Temperatures

140 °C / 2h



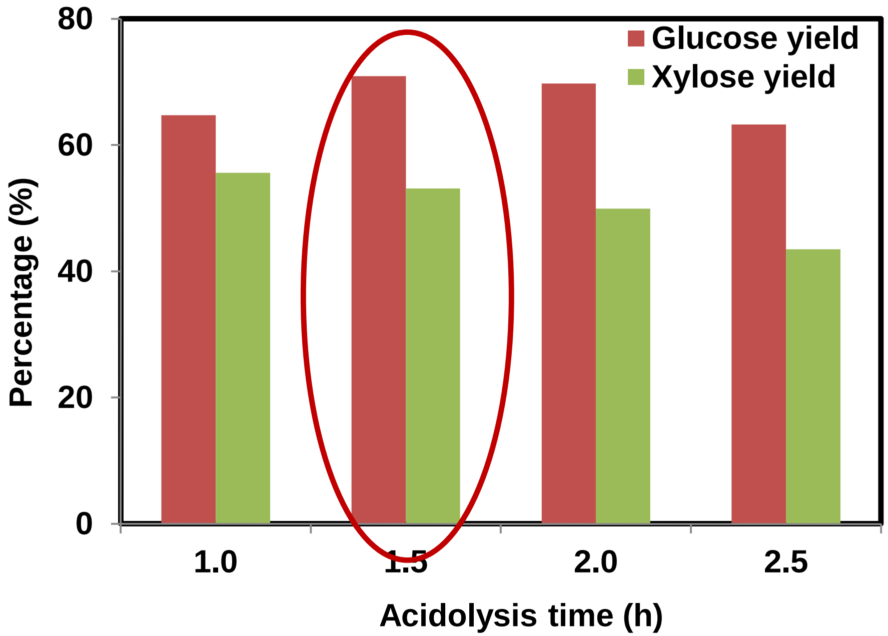
150 °C / 2h



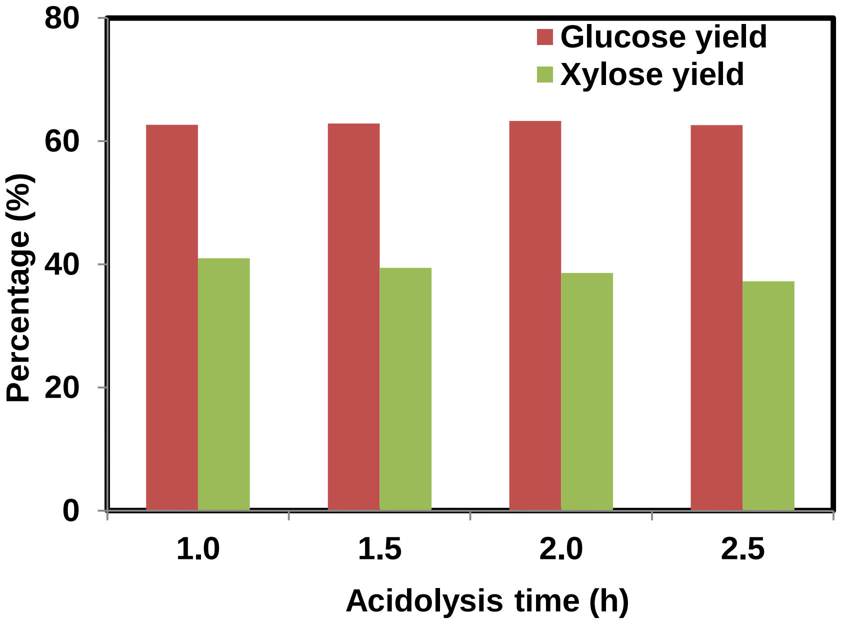
- With increased temperature, higher glucose yields and lower xylose yields were obtained after acidolysis indicating xylose degradation

Sugar Yields with Different Solid Loadings

160 °C / 10% solid



160 °C / 15% Solid



- After PT at 160 °C/2 h, 15% solid loading gives comparable glucose yields to 10%
- ***Maximum 71% glucose was obtained*** which is the highest glucose yield obtained in 10 L scale

Sugar Yields with Different ILs

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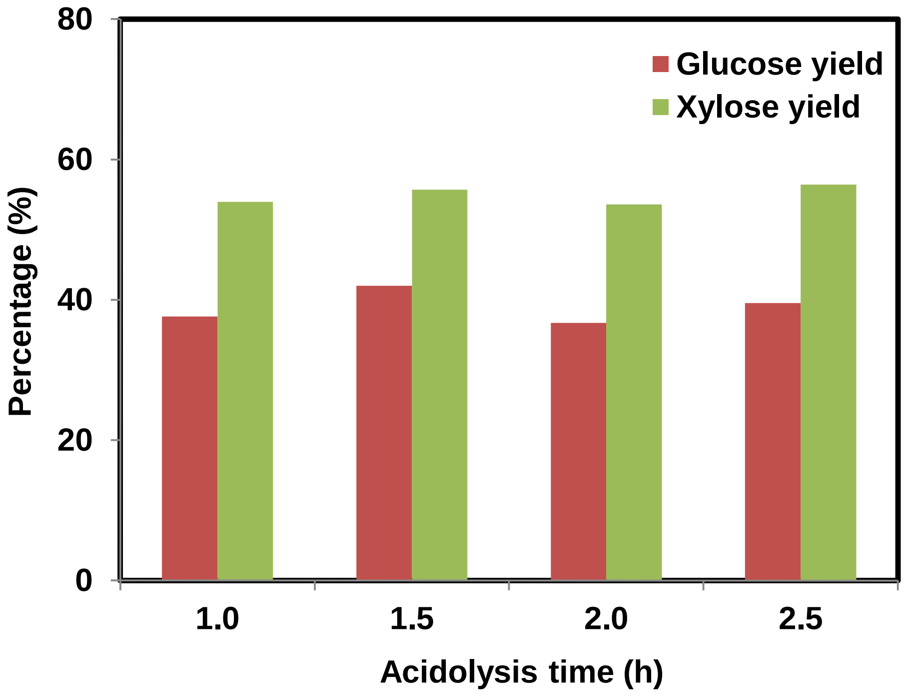
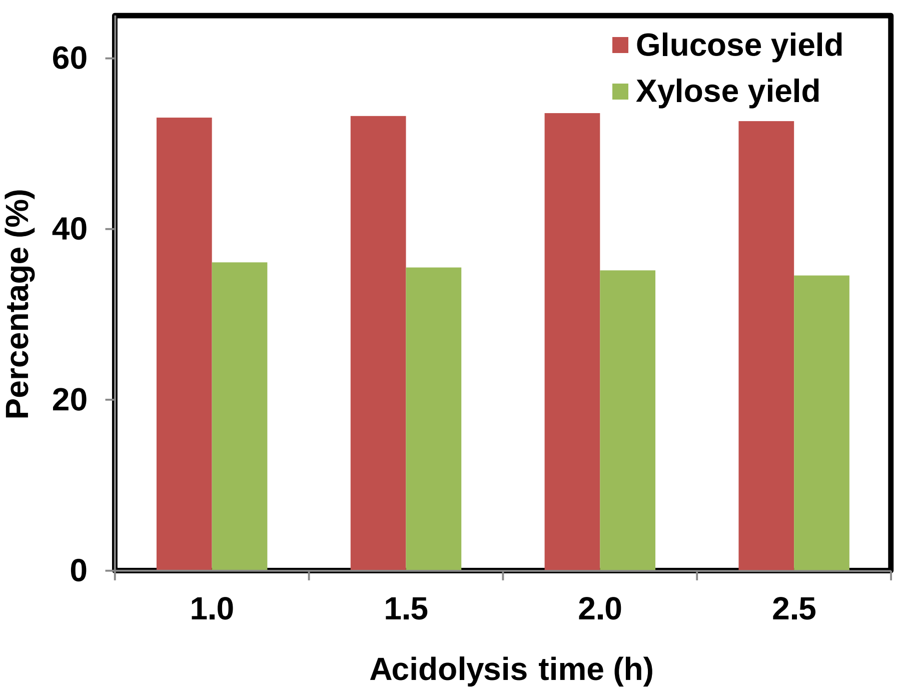
$[C_4C_1mim]Cl$



$[C_2C_1mim]Cl$



- After PT at 140 °C/2 h, $[C_2C_1mim]Cl$ results in higher glucose yield compared to $[C_4C_1mim]Cl$

Run 10 Mass and Energy Balance

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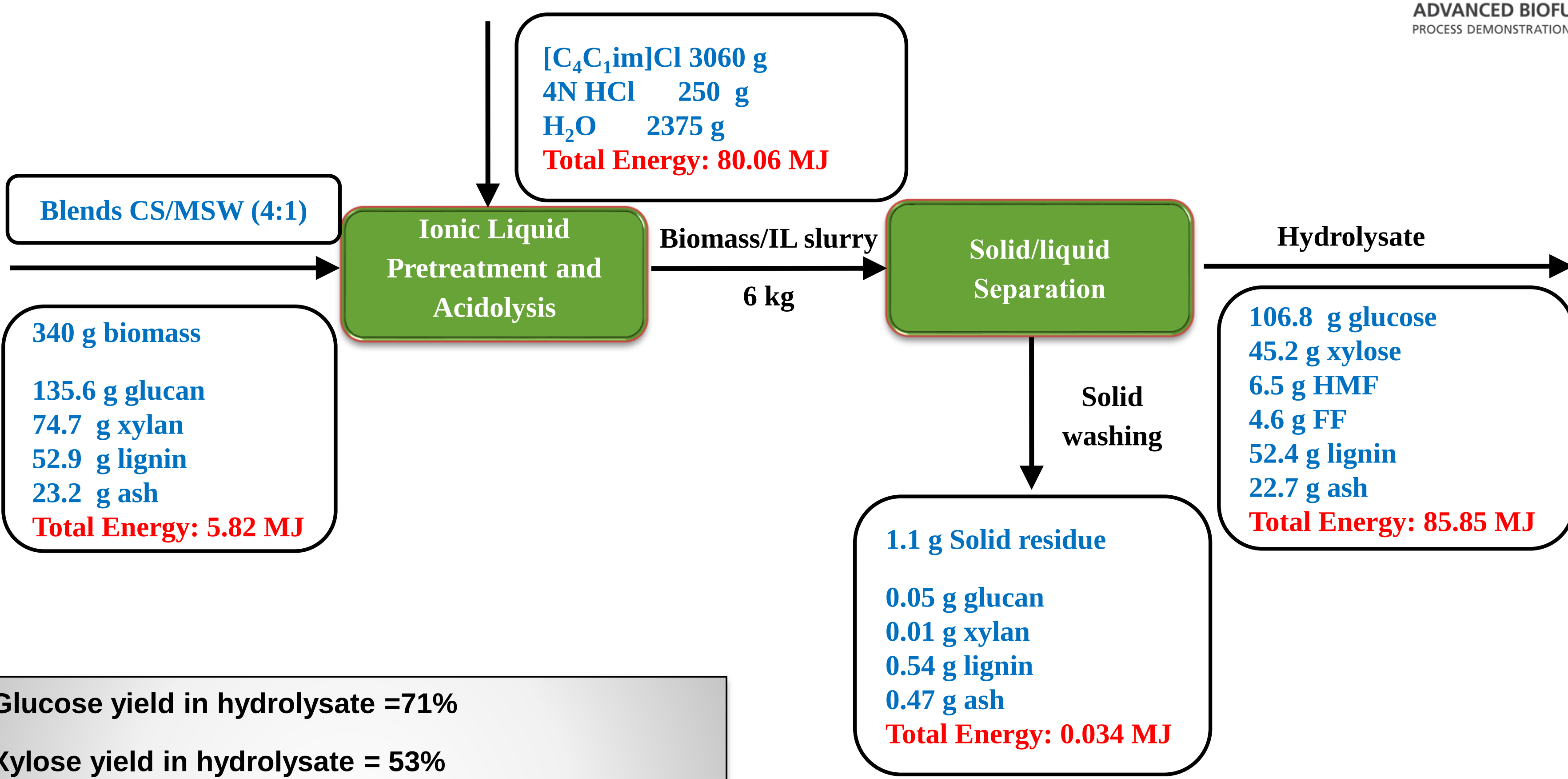

Glucose yield in hydrolysate =71%

Xylose yield in hydrolysate = 53%

Overall glucose balance = 81%

Overall xylose balance = 67%

Overall lignin recovery from solid stream = 1%

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Run 11 Mass Balance

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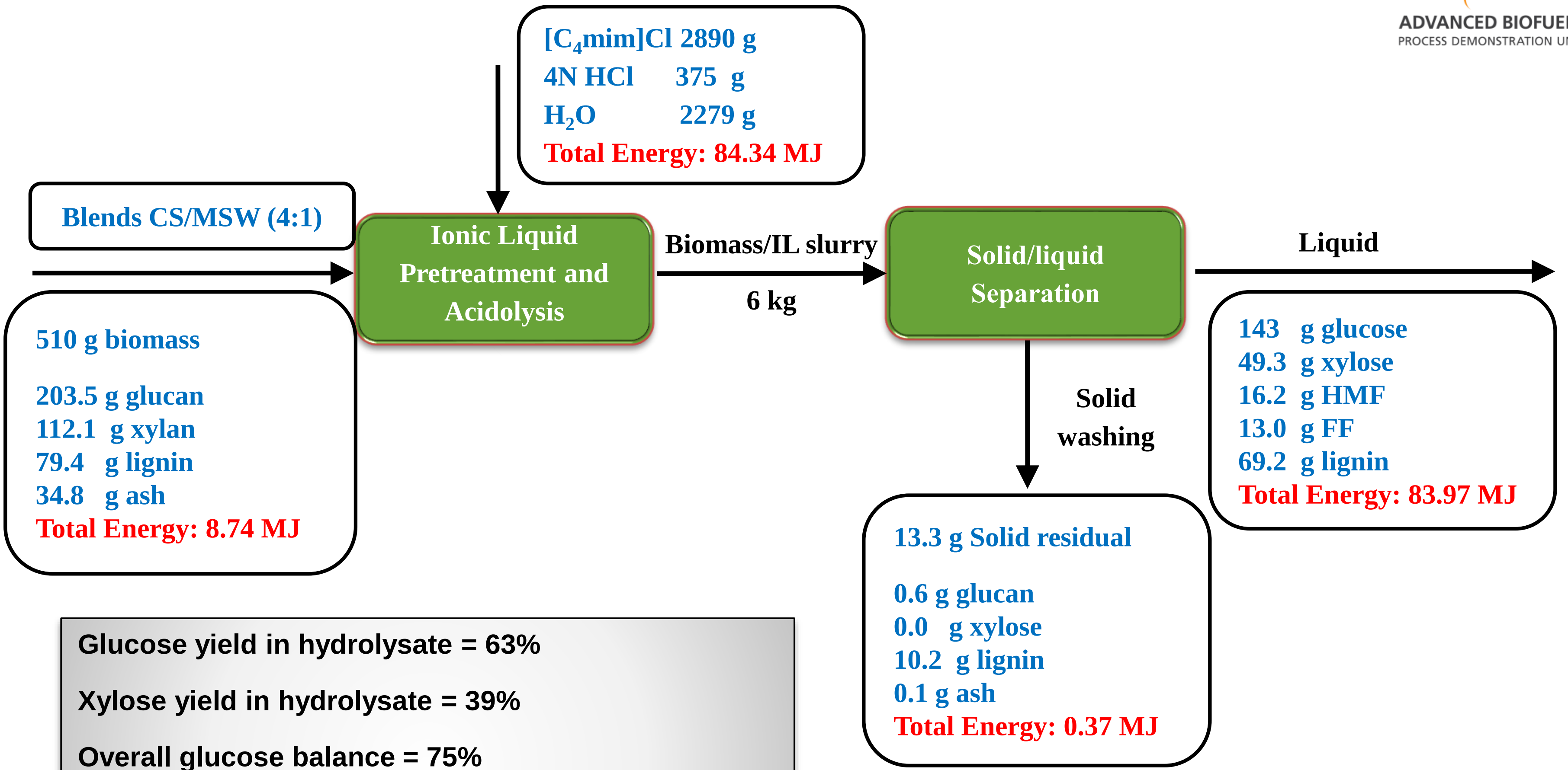

Glucose yield in hydrolysate = 63%

Xylose yield in hydrolysate = 39%

Overall glucose balance = 75%

Overall xylose balance = 56%

Overall lignin recovery from solid stream = 13%

Summary - Key Findings

- Developed an integrated process for IL based deconstruction technologies.
- Successfully demonstrated 200-fold scale up of MSW/CS blends IL acidolysis.
- Optimized conditions in tube reactor at SNL cannot be applied directly to the 10 L Parr vessels due to the different reactor configurations.
- The scale up attempt and process integration will leverage the opportunity towards a cost-effective sugar/lignin production technology.