Successful Solid-Liquid Separations Within a Biomass-to-Ethanol Process

June 19, 2003
Andy Aden
National Renewable Energy Laboratory
• Only national laboratory dedicated to renewable energy and energy efficiency R&D
• Research spans fundamental science to technology solutions
• Collaboration with industry and university partners is a hallmark
• Research programs linked to market opportunities
Energy Security
- Dramatically reduce or even end dependence on foreign oil
  - Biomass is the only renewable that directly reduces or dependency on liquid transportation fuels

Economics
- Spur the creation of a domestic bioindustry
  - The new industrial biorefinery model, with its production of products including fuels and chemicals from biomass, will help enable this domestic industry

Environment
- Carbon Neutral Processes
- Lower GHG emissions

Part of the answer in other EERE Programs/Interest
- Biomass to Hydrogen and Distributed Energy
The New Industrial Biorefinery

**Biomass Feedstock**
- Trees
- Grasses
- Agricultural Crops
- Agricultural Residues
- Animal Wastes
- Municipal Solid Waste

**Conversion Processes**
- Enzymatic Fermentation
- Gas/liquid Fermentation
- Acid Hydrolysis/Fermentation
- Gasification
- Combustion
- Co-firing

**USES**

**Fuels:**
- Ethanol
- Renewable Diesel

**Power:**
- Electricity
- Heat

**Chemicals**
- Plastics
- Solvents
- Chemical Intermediates
- Phenolics
- Adhesives
- Furfural
- Fatty acids
- Acetic Acid
- Carbon black
- Paints
- Dyes, Pigments, and Ink
- Detergents
- Etc.

**Food and Feed**
Biomass’ Major Molecules

**Lignin: 15-25%**
- Complex network of aromatic compounds
- High energy content
- Treasure trove of novel chemistry

**Hemicellulose: 23-32%**
- A collection of unusual 5- and 6-carbon sugars linked together in long, substituted chains
- Xylose is the 2nd most abundant sugar in biosphere

**Cellulose: 38-50%**
- Long chains of glucose
- Most abundant form of carbon in biosphere
Solid-liquid Separations

**Conditioning:**
- 1) Acid Recovery (washing)
- 2) Detoxification / gypsum recovery

**Dewatering:**
- drying lignin residue for combustion
- liquor sent to evaporation to recover dissolved solids, recycled

http://www.nrel.gov/docs/fy02osti/32438.pdf
Biomass Separation Challenges

- Porosity / Structural stability
  - Structure behaves “sponge-like” taking up water, but not readily releasing it

- High temperature environment
  - Corrosive
  - Potentially reactive

- Other materials present
  - Oils
  - Fines
  - Proteins

- Lack of system characterization
  - Particle size distribution
NREL engaged the Harris Group (HGI) to perform vendor testing, process design, and costing for S/L sep

- NREL Subcontract ACO-9-29067-01
  http://www.afdc.doe.gov/pdfs/5455.pdf

Several equipment types explored

- Hydrolysate wash: horizontal belt filter, pressure filter
- Dewatering: centrifuge, filter press, belt filter press, pressure filter

Results incorporated into process design and techno-economic analysis for 2000 dry metric tonnes per day
## Subcontract Results

### Hydrolysis

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Manufacturer</th>
<th>Wash Water (lb/lb feed)</th>
<th>Acetic Acid Residual (mg/mL)</th>
<th>Estimated Capital Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Horizontal belt</td>
<td>“B”</td>
<td>1.1 – 1.2</td>
<td>No data</td>
<td>$6,300,000</td>
</tr>
<tr>
<td>filter</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Horizontal belt</td>
<td>“D”</td>
<td>1.1</td>
<td>1.7</td>
<td>$27,000,000</td>
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<tr>
<td>filter</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Pressure filter</td>
<td>“C”</td>
<td>0.58</td>
<td>0.9</td>
<td>$8,500,000</td>
</tr>
</tbody>
</table>

### Post-Distillate

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Manufacturer</th>
<th>Cake Solids</th>
<th>Estimated Capital Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centrifuge</td>
<td>“A”</td>
<td>19.9%</td>
<td>$8,800,000</td>
</tr>
<tr>
<td>Centrifuge</td>
<td>“B”</td>
<td>22.9%</td>
<td>no data</td>
</tr>
<tr>
<td>Filter press, Opt 1</td>
<td>“B”</td>
<td>34.9% - 39.7%</td>
<td>$17,300,000</td>
</tr>
<tr>
<td>Filter press, Opt 2</td>
<td>“B”</td>
<td>39.7% - 44.4%</td>
<td>$24,500,000</td>
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<tr>
<td>Pressure filter</td>
<td>“C”</td>
<td>88.0%*</td>
<td>$8,100,000</td>
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<tr>
<td>Belt filter press</td>
<td>“B”</td>
<td>no data</td>
<td>no data</td>
</tr>
</tbody>
</table>

*This seemed abnormally high. After re-analyzing the data, 56% solids used in design.*
Do These Results Make Sense?

- From AIChE

Solid/Liquid Separation:
Course #289

<table>
<thead>
<tr>
<th>CAKE FILTERS</th>
<th>Solid Dryness</th>
<th>Liquid Clarity</th>
<th>Thickening</th>
<th>Washing</th>
<th>Classification</th>
<th>Particle Breakage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravity filters</td>
<td>E</td>
<td>G</td>
<td>P</td>
<td>G</td>
<td>-</td>
<td>E</td>
</tr>
<tr>
<td>Batch, semi-batch</td>
<td>G</td>
<td>G</td>
<td>P</td>
<td>E</td>
<td>-</td>
<td>E</td>
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<tr>
<td>Vacuum filters</td>
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<tr>
<td>Continuous vacuum filters</td>
<td>E</td>
<td>E</td>
<td>P</td>
<td>F</td>
<td>-</td>
<td>G</td>
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<tr>
<td>Batch, semi-batch pressure filters</td>
<td>E</td>
<td>E</td>
<td>P</td>
<td>F</td>
<td>-</td>
<td>G</td>
</tr>
<tr>
<td>Continuous pressure filters</td>
<td>E</td>
<td>E</td>
<td>P</td>
<td>F</td>
<td>-</td>
<td>G</td>
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<td>Compression filters</td>
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<td>F</td>
<td>-</td>
<td>F</td>
<td>-</td>
<td>P</td>
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<tr>
<td>Filtering centrifuges</td>
<td>G</td>
<td>E</td>
<td>P</td>
<td>E</td>
<td>-</td>
<td>P-P</td>
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<tr>
<td>Precoat filters</td>
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<td>E</td>
<td>-</td>
<td>-</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>SETTLELS</th>
<th>Solid Dryness</th>
<th>Liquid Clarify</th>
<th>Thickening</th>
<th>Washing</th>
<th>Classification</th>
<th>Particle Breakage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravity - clarifier</td>
<td>P</td>
<td>E</td>
<td>G</td>
<td>-</td>
<td>-</td>
<td>E</td>
</tr>
<tr>
<td>Gravity - thickener</td>
<td>P</td>
<td>G</td>
<td>E</td>
<td>P</td>
<td>-</td>
<td>E</td>
</tr>
<tr>
<td>Tubular centrifuge</td>
<td>P</td>
<td>E</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>F</td>
</tr>
<tr>
<td>Disc centrifuge - solid bowl</td>
<td>P</td>
<td>E</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>G</td>
</tr>
<tr>
<td>Disc centrifuge - desludger</td>
<td>P</td>
<td>E</td>
<td>E</td>
<td>P</td>
<td>-</td>
<td>F</td>
</tr>
<tr>
<td>Disc centrifuge - nozzle</td>
<td>P</td>
<td>G</td>
<td>G</td>
<td>F</td>
<td>P</td>
<td>F</td>
</tr>
<tr>
<td>Solid bowl centrifuge</td>
<td>F-G</td>
<td>E-G</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
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<tr>
<td>Hydrocyclones</td>
<td>P</td>
<td>P</td>
<td>G</td>
<td>F</td>
<td>E</td>
<td>P</td>
</tr>
</tbody>
</table>

E - excellent, G - good, F - fair, P - poor, “-” - should not be considered
NREL’s Process Development Unit

A fully integrated biomass to ethanol plant

Processes one ton per day of biomass  Capable of round the clock operations
Produces 100 proof ethanol  Collects data from over 900 process points
Flexible integration and set-up  Batch and continuous operations
PDU S/L Separation Equipment

- **Pilot-scale equipment:**
  - Bock lined centrifuge
  - Sharples solid bowl decanter centrifuge
  - Pneumapress pressure filter
  - Bench-scale testing also

- **Current uses:**
  - In-house program research
  - WFO/CRADA with industrial partners
  - Hydrolysate / Fermentation residue production for testing
Pressure Filter Data

<table>
<thead>
<tr>
<th>Number of Washes</th>
<th>Wash Ratio (kg water/kg slurry)</th>
<th>Wash Ratio (kg water/kg BD solids)</th>
<th>Xylose Recovered in Filtrate (% initial)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>45</td>
</tr>
<tr>
<td>1</td>
<td>0.67</td>
<td>2.67</td>
<td>57</td>
</tr>
<tr>
<td>1</td>
<td>0.73</td>
<td>2.91</td>
<td>71</td>
</tr>
<tr>
<td>1</td>
<td>0.91</td>
<td>3.62</td>
<td>65</td>
</tr>
<tr>
<td>2</td>
<td>1.09</td>
<td>4.34</td>
<td>98</td>
</tr>
<tr>
<td>2</td>
<td>1.09</td>
<td>4.34</td>
<td>97</td>
</tr>
<tr>
<td>2</td>
<td>0.87</td>
<td>3.46</td>
<td>96</td>
</tr>
<tr>
<td>2</td>
<td>0.58</td>
<td>2.31</td>
<td>96</td>
</tr>
</tbody>
</table>

Pilot Scale Test—Sunds Pretreated Corn Stover (NREL)

<table>
<thead>
<tr>
<th>Number of Washes</th>
<th>Wash Ratio (kg water/kg BD solids)</th>
<th>Wash Ratio (kg water/kg BD solids)</th>
<th>Xylose Recovered in Filtrate (% initial)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>71</td>
</tr>
<tr>
<td>1</td>
<td>0.43</td>
<td>4.97</td>
<td>86</td>
</tr>
<tr>
<td>1</td>
<td>0.66</td>
<td>7.70</td>
<td>90</td>
</tr>
<tr>
<td>1</td>
<td>1.56</td>
<td>18.12</td>
<td>92</td>
</tr>
</tbody>
</table>
“Hot Wash” Process Variation

- Adding a pressurized “hot wash” step immediately following pretreatment (while still at T&P) improves enzymatic digestibility

- Theorize that lignin re-precipitates back onto cellulose, interfering w/ enzyme hydrolysis

- Pneumapress was only vendor in subcontract with suitable equipment for elevated temperature (135°C) operation

- Soluble lignins with potentially valuable unique properties are separated out

NREL Patents #6,022,419 & #6,228,177
What’s Next?

- Working with industrial partners.
- Working with different feedstock with different S/L characteristics.
- Different co-product opportunities.
- Looking to improve economics for downstream processing.
This work supported by the Office of the Biomass Program of the U.S. Department of Energy Office of Energy Efficiency and Renewable Energy

Harris Group Inc.