Process Improvement to Biomass Pretreatment for Fuels and Chemicals

March 24th 2015
Technology Area Review: Biochemical Conversion

Principal investigator: Farzaneh Teymouri
Organization: MBI, Lansing MI
Tel.: (517) 337-3181, www.mbi.org

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Goal Statement

Feedstock supply, including logistics systems and sustainable high quality feedstock, inadequate supply chain infrastructure, and feedstock cost are among the critical barriers that have been identified by DOE and EERE for commercialization of cellulosic biofuels and chemicals in the United States.

One of the leading concepts for addressing the feedstock logistics challenge is the relocation of preprocessing and pretreatment operations closer to biomass feedstock harvest locations through a system of Regional Biomass Processing Depots (RBPDs).

*An inexpensive pretreatment, suitable to a wide variety of feedstocks and fermentation systems, is essential to enable the RBPD concept and achieve the commercial goals.*
Quad Chart Overview

Timeline

• Start date: September 01, 2011
• Project end date: February 28, 2015
• 100% complete

Barriers

• Bt-E Pretreatment cost
• Bt-K Biological process integration

Partners

• Michigan State University (MSU)
• Idaho National Lab (INL) (involvement: 14%)

Budget

<table>
<thead>
<tr>
<th></th>
<th>Total Costs FY 10 – FY 12</th>
<th>FY 13 Costs</th>
<th>FY 14 Costs</th>
<th>FY 15 Costs</th>
<th>Total Planned Budget</th>
</tr>
</thead>
<tbody>
<tr>
<td>DOE Funded</td>
<td>$924,626</td>
<td>$1,795,636</td>
<td>$914,919</td>
<td>$41,763</td>
<td>$3,676,944</td>
</tr>
<tr>
<td>MBI Cost Share</td>
<td>$544,405</td>
<td>583,340</td>
<td>($19,443)</td>
<td>$3,839</td>
<td>$1,069,254</td>
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<tr>
<td>MSU cost share</td>
<td>$29,922</td>
<td>$50,215</td>
<td>$56,000</td>
<td>$136,137</td>
<td></td>
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</tbody>
</table>

**Project Management Team**: Farzaneh Teymouri, Bernie Steele, and Tim Campbell, all of MBI.

**Feedstock Team**: Kevin Kenney of INL, supported by Tyler West and David Thompson.

**AFEX Process Improvement Team** is led by Tim Campbell MBI, supported by Bruce Dale of MSU, Richard Hess of INL.

**Modeling Team** is led by Bryan Bals of MBI, supported by Bruce Dale of MSU, Farzaneh Teymouri and Tim Campbell of MBI, Kevin Kenny of INL.

**Commercialization Team** is led by Allen Julian of MBI.
Project Overview
History
How can we:

• Upgrade raw biomass to make sugars more accessible?
• Handle, store, transport low-density biomass?
• Establish upgraded biomass as a tradable commodity?
Solution: Decentralized Preprocessing and Pretreatment

Regional Biomass Processing Depots (RBPDDs): Convert regional, distinct biomass sources into dense, stable, shippable intermediate commodities

Image courtesy of INL (www.inl.gov)
AFEX Biomass Pretreatment
Promising option for RBPDs concept
Ammonia Fiber Expansion (AFEX)

Raw Biomass

Reaction
- Moist biomass is contacted with ammonia
- Temperature and pressure are increased
- Contents soak for specified time at temperature and ammonia load
- Pressure is released
- Ammonia is recovered and reused

Expansion

Densification

Treated Biomass

Ammonia Recovery

AFEX Pellets
- AFEX pellets 9-fold denser than biomass
- Stable, storable, readily transportable
Reactor designs for AFEX

**AFEX 1**
- Initial design created in early 2000s
- Based on Pandia-type reactors
- Continuous treatment process
- High capital cost, desirable for high (1000+) tons/day

**AFEX 3**
- Vertical packed bed batch reactors in pairs
- Ammonia recovered directly within beds
- Low capital, simple design suitable for small (100) tons/day
AFEX 3 – Packed Bed AFEX

Replacing Pandia-type reactor with a new design that is simpler in equipment requirements and operation and incorporates ammonia recovery and reuse directly in the reactor.

**Concept**
Treat moist, ground biomass in packed beds, using **five steps**:

1. **Pre-steam**
   - Steam
   - Air

2. **NH₃ Charge**
   - NH₃ (vapor)

3. **Soak**

4. **Depressurize**
   - NH₃ (vapor)

5. **Steam Strip**
   - NH₃ (vapor)

Diagram:
- Step 1: Pre-steam with steam and air.
- Step 2: NH₃ Charge with NH₃ (vapor).
- Step 3: Soak.
- Step 4: Depressurize with NH₃ (vapor).
- Step 5: Steam Strip with NH₃ (vapor).
Proof of Concept

Laboratory skid

- Installed August 2010
- Bed dimensions (3):
  - 3.9 inch D X 48 inch L
  - 0.5 – 1.0 kg biomass per bed
- Demonstrated good results with:
  - Corn stover
  - Wheat straw
  - Oat hulls
  - Switchgrass
- ≥ 98% NH$_3$ recovery
- Benchmark for project
1-Project Objectives

• Reduce (>50%) the capital cost at commercial scale Regional Biomass Processing Depots (RBPDs) (100 tons per day) compared to AFEX 1 by:
  – Altering the AFEX pretreatment system design to exploit the physical and chemical characteristics of the ammonia catalyst and enable:
    • Improved ammonia loading and activity efficiency
    • Improved biomass transfer efficiency within the system
    • Improved ammonia recovery and reuse efficiency

• Reduce the cost of production of ethanol by 16% by using AFEX 3 design instead of the AFEX 1 design in RBPDs
Technical Approach

- Scale up packed bed reactor by factor of 50
- Compare performance with benchmarks
- Meet the following critical success factors:

<table>
<thead>
<tr>
<th>Critical success factors</th>
<th>Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sufficient throughput for commercial-scale reactor (19.5 m³):</td>
<td>Intermediate target</td>
</tr>
<tr>
<td>- Biomass bed density</td>
<td>Final Target</td>
</tr>
<tr>
<td>- Cycle time per pair of reactor</td>
<td>17 tons/reactor/day</td>
</tr>
<tr>
<td></td>
<td>25 tons/r/d</td>
</tr>
<tr>
<td></td>
<td>80 kg/m³</td>
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<tr>
<td></td>
<td>100 kg/m³</td>
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<tr>
<td></td>
<td>120 min</td>
</tr>
<tr>
<td></td>
<td>110 min</td>
</tr>
<tr>
<td>Efficacy of AFEX3 engineering scale pretreatment as verified by</td>
<td>Sugar yield ≥ conventional</td>
</tr>
<tr>
<td>sugar yields</td>
<td>AFEX (&gt;75% of available</td>
</tr>
<tr>
<td></td>
<td>sugars)</td>
</tr>
<tr>
<td>Efficient NH3 recovery and reuse</td>
<td>98% ammonia recovery</td>
</tr>
<tr>
<td>Meet targeted cost reduction of pretreatment</td>
<td>Capital cost reduction</td>
</tr>
<tr>
<td></td>
<td>Intermediate target</td>
</tr>
<tr>
<td></td>
<td>Final Target</td>
</tr>
<tr>
<td></td>
<td>30%</td>
</tr>
<tr>
<td></td>
<td>50%</td>
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</table>
Management Approach

**Task A.** Determine the effects of feedstock specifications and reactor design on pretreatment efficacy and ammonia recycle at lab scale.

**Success measure:** >95% ammonia recovery and >70% sugar yields at high solid loading

**Task B.** Preparation of biomass for engineering scale AFEX 3

**Deliverable:** Preprocess about 20 tons of corn stover at spec (particle size, shape, and moisture)

**Task C.** Design and fabrication of engineering scale AFEX 3

**Deliverable:** Install a complete AFEX 3 system with capacity of processing at least 30 kg of corn stover per reactor bed

**Task D.** Process improvement development at engineering scale

**Success measure:** Reach target ammonia recovery ≥98%, show equivalent hydrolysis yield for corn stover treated in the AFEX 3 system compared to the corn stover treated in lab scale reactor

**Task E.** Generate and update techno-economic models of the biomass-to-fuel process

**Deliverable:** Design process flow diagram, material and energy flow, TEC models for production of ethanol from both AFEX 1 and AFEX 3 system

**Targets:** Intermediate = 30% reduction in CAPX and OPEX of AFEX

Final = 50% reduction in CAPX and OPEX of AFEX

**Task F.** Determine the quality of pretreated biomass through fermentation use tests

**Success measure:** Converting >95% of glucose and >85% of xylose generated from AFEX treated biomass to ethanol
3-Techincal Accomplishments
Progress/Results
Progress reported in the last review
Task A. Addressing Potential Risks Using Lab Scale AFEX 3

Determine the effects of feedstock specifications and reactor design on pretreatment efficacy and ammonia recycle at lab scale.

**Targets:** 95% ammonia recovery and >70% sugar yields at high solid loading

**Accomplishments:**
- Reached target bed density: 100 kg/m$^3$ by using the baskets
- Determined suitable specifications for biomass
- Determined reactor orientation and aspect ratio to achieve ammonia recovery target

Able to recycle almost pure NH$_3$
Task B. Biomass Procurement (INL)

Corn stover bales (25 tons) at INL

Grinding Corn stover at INL

Transferring to the supersacks

Supersacks stored indoors
Biomass Preparation

Supersacks containing biomass

Biomass unloading building

Moisture adjustment in blender

Basket packer

Compressed into baskets, density ~100 kg/m³
Task C. Design of Engineering Scale AFEX 3
AFEX Pilot – Valve skid, COMP

Valve skid:
• Valves –
  • 60 total, 20 actuated
• Instruments –
  • 12 temp, 12 pressure, 3 flow
• Heat exchangers –
  • NH₃ Evaporator, condenser

Compressor:
• Rotary screw (Frick RXF-15)
• Suction 0, discharge 300 psig
• NH₃ displacement ≈ 3 kg/min
Task C. Installation of AFEX3 System

Installation started in Mid February 2013 and was completed by Mid March 2013.

Accomplishments/ major milestone:

Engineering scale AFEX3 capable of treating more than 35 kg of biomass per bed was designed, fabricated and installed.

Engineering scale AFEX3 system installed in MBI building.
Progress made since the last review
Task D. Operation of AFEX3 pilot scale system

More than 480 beds treated using the five step cycle:

Step 1: Pre-steam
Step 2: NH₃ Charge
Step 3: Soak
Step 4: Depressurize
Step 5: Steam Strip
AFEX Pilot Plant – Treatment Cycle

Bed Temperatures, Pressure

- 1) Presteam
- 2) NH₃ charge
- 3) Soak
- 4) Depressurize
- 5) Steam strip

Cool

Temperature (°C)

Pressure (psig)

Time (minutes)
AFEX Pilot Plant – Treatment Cycle
Bed Temperatures, Pressure

Time (minutes)

Temperature (°C)
Pressure (psig)

Lead Bed

Lag Bed

Top Temp
Bottom Temp
Pressure

mbi
AFEX Pilot Plant – NH3 Balance (early 2014)

<table>
<thead>
<tr>
<th></th>
<th>NH₃ Transfer (kg)</th>
<th>NH₃ Recovery (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current</td>
<td>14</td>
<td>70</td>
</tr>
<tr>
<td>Recoverable</td>
<td>&gt;19</td>
<td>&gt;95</td>
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</tbody>
</table>

-20.0 kg charge

LEAD BED

-0.6 kg

CONDENSER

-0.7 kg

COMP

MANIFOLD

-0.9 kg

V/L SEP

-3.8 kg condensate

LAG BED

+6.0 kg charge
Steam Stripping Performance
Pilot vs. Lab Scale

Vapor Composition (mol% NH$_3$) vs. Free NH$_3$ removal (mass%)

- Lab scale
- Pilot scale
AFEX-Treated Corn Stover
Enzyme Hydrolysis Results

Glucose yield (% theor)

Xylose yield (% theor)

Solid line: Average of all the runs
Dashed line: Benchmark performance
May 2014:
- Shaft seal leak
- Bearing cages disintegrated
- Heavy corrosion and scaling throughout machine
- Cause: H2O accumulation
## AFEX 3 Pilot Plant - Compressor Issues

<table>
<thead>
<tr>
<th>Issue</th>
<th>Causes</th>
<th>Problems</th>
<th>Solutions</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₂O accumulation</td>
<td>• Stop/start duty</td>
<td>• Corrosion</td>
<td>• Hot gas bypass</td>
</tr>
<tr>
<td></td>
<td>• Mist formation in condenser</td>
<td>• Oil degradation</td>
<td>• Mist eliminator</td>
</tr>
<tr>
<td>Overheating</td>
<td>Control valve malfunction</td>
<td>Load inhibition at high discharge T</td>
<td>Proper control valve settings</td>
</tr>
<tr>
<td>Compressor wrong approach for AFEX?</td>
<td>Compressor control incompatible with batch process?</td>
<td>• Continued H₂O accumulation</td>
<td>Develop absorption approach</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Others?</td>
<td></td>
</tr>
</tbody>
</table>
AFEX Bed-to-Bed NH3 Transfer

Hot Gas Bypass line installed Oct 2014

- Allows continuous operation between bed-to-bed NH₃ transfers
- Compressor maintains operating temp 220°F
- Oil P drop problem
- High electrical load

• Investigating alternative approach for ammonia recovery – remains ongoing
AFEX Pilot Plant – Current NH3 Balance

<table>
<thead>
<tr>
<th></th>
<th>NH₃ Transfer (kg)</th>
<th>NH₃ Recovery (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Validation Run</td>
<td>20</td>
<td>82</td>
</tr>
<tr>
<td>Recoverable</td>
<td>23</td>
<td>94</td>
</tr>
</tbody>
</table>

LEAD BED

+24.5 kg charge

-1.8 kg

CONDENSER

COMP -0.4 kg

MANIFOLD -0.9 kg

V/L SEP

-1.4 kg condensate

LAG BED

+4.4 kg charge
AFEX Treatment Cycle

**Single Reactor:** cycle time based on pilot plant results

<table>
<thead>
<tr>
<th>Steps</th>
<th>NH$_3$ Charge</th>
<th>Makeup NH$_3$ Add $\rightarrow$ Soak</th>
<th>DePressurize $\rightarrow$ Steam Strip</th>
<th>Unload $\rightarrow$ Reload $\rightarrow$ PreSteam</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time (minutes)</td>
<td>19</td>
<td>36</td>
<td>19</td>
<td>36</td>
</tr>
</tbody>
</table>

Reactor Cycle Time = 110 minutes

**Reactor Pair:** 24 hours of operation

**Depot-Scale Reactor Pair Output:** $V_{\text{Bed}} = 19.4 \text{ m}^3$; $\rho_{\text{Bed}} = 100 \text{ kg(dry)/m}^3$

1,940 kg dry biomass / Bed

26 Beds / Pair / Day

$\{ \} = 50 \text{ tonne dry biomass / Pair / Day}$
Task E. Depot design and Techno economic analysis

Depot feedstock ≈ 100 TPD baled corn stover

- Reactors: (4) 5 ft Ø x 35 ft H
- One compressor

100 TPD AFEX depot CAD drawing by Corrie Nichol, INL
AFEX Depot

$9.5 million capital investment

100 TPD AFEX depot top view drawing by Corrie Nichol, INL
## Depot cost analysis

<table>
<thead>
<tr>
<th>Item</th>
<th>Cost ($/metric dry ton)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capital cost + Return on Investment</td>
<td>$23.25</td>
</tr>
<tr>
<td>Labor</td>
<td>$17.07</td>
</tr>
<tr>
<td>Electricity</td>
<td>$13.65</td>
</tr>
<tr>
<td>Steam for AFEX</td>
<td>$4.90</td>
</tr>
<tr>
<td>Heat for Drying</td>
<td>$7.08</td>
</tr>
<tr>
<td>Maintenance</td>
<td>$5.24</td>
</tr>
<tr>
<td>Ammonia</td>
<td>$28.14</td>
</tr>
<tr>
<td>Corn Stover</td>
<td>$63.85</td>
</tr>
<tr>
<td><strong>Subtotal (Depot costs)</strong></td>
<td><strong>$99.33</strong></td>
</tr>
<tr>
<td><strong>Subtotal (Operating costs)</strong></td>
<td><strong>$139.93</strong></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>$163.18</strong></td>
</tr>
</tbody>
</table>

Total cost of pellets is ~$160/DMT

Cost determined by:
- Quotes for major equipment (AFEX reactors, dryer, pelletizer, grinders)
- INL estimates for biomass handling
- MBI estimates for ammonia recovery cost
- Pilot scale data for steam use and exiting moisture
- MBI and INL estimates for electricity use for all equipment
- 46% capital cost reduction compared to AFEX 1
- 29% Total cost reduction compared to AFEX 1
Task F. Testing Fermentability of Sugars from AFEX 3 Treated Biomass (MBI and MSU)

- In stirred tank reactor (2500 l)
- Biomass: pellets made from corn stover treated in AFEX 3 pilot reactor.
- SHF fermentation using Z. mobilis (utilizing both C5 and C6)
- Process started with 22% solid loading
- Pellets were used as is, no sterilization
- No sign of contamination
- Did not remove unhydrolyzed residue prior to inoculation
- Complete utilization of both glucose and xylose.

Accomplishment:
Confirmed the fermentability of generated sugar

- Using NREL 2011 model and AFEX depots, ethanol production costs $2.26/gal at $65/DMT corn stover, equivalent to $3.23/gal gasoline equivalent assuming ammonia recovery obtained in pilot scale
- Assuming an improved ammonia recovery, $3.10/gal gasoline equivalent can be obtained
- MBI is collaborating with Novozymes to further decrease the enzyme loading during hydrolysis

Feedstock supply, including logistics systems and sustainable high quality feedstock, inadequate supply chain infrastructure, and feedstock cost are among the critical barriers that have been identified by DOE and EERE for commercialization of cellulosic biofuels and chemicals in the Unites States.

- AFEX treated corn stover pellets are compatible with corn grain supply and logistics infrastructure and can be stored and transported long distances
- AFEX treatment enables the “hub and spoke” model identified by DOE as a potential solution to the critical barrier of feedstock supply and logistics
Relevance - Impact beyond the scope of the project

• DOE support enabled following:
  – Demonstrated pilot scale pretreatment and pelletization of corn stover
  – Demonstrated 2500 L hydrolysis and fermentation of pellets
  – Successful exploratory feeding trials for both beef and dairy cattle

• Successful completion of project is enabling:
  – Ongoing FDA approval efforts for cattle feed application
  – Currently engaged with EPC firm on depot design
Summary

1) Approach
   1) Reduce the capital cost of AFEX pretreatment by designing, building, and operating an engineering scale packed bed AFEX reactor system

2) Technical accomplishments
   1) Designed, fabricated, and installed engineering scale AFEX 3 reactor
   2) Treated >450 beds of corn stover with no safety incidents
   3) Demonstrated ammonia recovery and sugar yields at pilot scale
   4) Model showing 46% capital cost reduction compared to previous design
   5) Demonstrated fermentability of pretreated biomass

3) Relevance
   AFEX 3 reactor will reduce cost of biofuel by reducing the capital cost of pretreatment at the depot scale

4) Critical Success factors and challenges
   Biomass throughput and ammonia recovery are key critical success factors to be addressed at the engineering scale

<table>
<thead>
<tr>
<th>Critical success factors</th>
<th>Target</th>
<th>Actual</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reactor Throughput</td>
<td>25 tons/reactor/day</td>
<td>25 tons/reactor/day</td>
</tr>
<tr>
<td>Sugar yields</td>
<td>&gt;75% of available sugars</td>
<td>75% at pilot</td>
</tr>
<tr>
<td>Ammonia Recovery</td>
<td>98% ammonia recovery</td>
<td>94% ammonia recovery</td>
</tr>
<tr>
<td>Capital Reduction</td>
<td>50%</td>
<td>46%</td>
</tr>
</tbody>
</table>
Publication and Presentation


Thompson DN, Campbell T, Bals B, Runge T, Teymouri F, Ovard LP (2013). Chemical preconversion: Application of low-severity pretreatment chemistries for commoditization of lignocellulosic feedstock. Biofuels, Accepted publication

Bals BD, Gunawan C, Moore J, Teymouri F, Dale BE. Enzymatic hydrolysis of pelletized AFEX™-treated corn stover at high solid loadings. Submitted to Biotechnology and Bioengineering.


Publication and Presentation


Cory Sarks, Bryan D. Bals, Mingjie Jin, Farzaneh Teymouri, Bruce E. Dale, and Venkatesh Balan. Fermentation condition optimization and economic analysis for ethanol production from pelletized AFEX™ corn stover using commercial enzymes and Zymomonas mobilis 8b. Manuscript submitted to Bioresource Technology
Commercialization Efforts

MBI’s vision is to make the AFEX technology available to the world on a low-cost, non-exclusive basis, so that its significant positive impacts on sustainable food, fuel, rural poverty, and the environment can be fully realized.

To achieve our vision, we must first take the technology from pilot to commercial (100 tons per day) scale, so that the technical and economic viability of the technology, as well as its positive societal impacts, can be demonstrated.

We are currently seeking partners who share our vision for the technology’s worldwide impact, and are willing to fund the construction of the “pioneer” AFEX depot and support the final stages of derisking the animal feed and biorefinery applications.

We are engaging with a leading engineering firm to complete the depot design and manage the procurement and construction phases.

Once proven in the pioneer depot, market forces will drive the propagation of the technology, with local farmers and entrepreneurs marshalling a combination of government, philanthropy, and investor support to build thousand of depots around the world.
Responses to Previous Reviewers’ Comments

• Economic viability is questionable
  – AFEX designed for depot – improved biomass logistics have overall cost benefits
  – NREL analysis suggests increasing size of biorefinery (only possible with pellets) leads to ~30 cent reduction in cost of biofuel
  – Possible benefits with retrofitting corn ethanol refinery
  – Current analysis suggests competitiveness with animal feed
  – Biofuel application is currently questionable but competitive with other technologies, but changes in energy market could lead to viability

• Concern with safety assessment
  – Ammonia used in refrigeration in similar quantities
  – >480 pilot runs with no safety incidents

• In July 2013 based on the second onsite validation visit a Go decision was made in the stage gate meeting.