

2013 DOE Bioenergy Technologies Office (BETO) Project Peer Review

Process Improvement to Biomass Pretreatment for Fuels and Chemicals

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Technology Area Review: Biochemical Conversion

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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.

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.

Our project is focused on reducing the cost of biomass treatment process through improvements to the unit operation of AFEX TM pretreatment.



Quad Chart Overview

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Timeline

- Start date: September 01, 2011
- Project end date: August 31, 2014
- 65% complete

Budget

DOE	FY 11: \$ 118,807 FY 12: \$ 1,425,650 FY 13: \$ 1,425,650
Cost Share	FY 11: \$ 29,704 FY 12: \$ 356,414 FY 13: \$ 356,414
Years Funded	9/1/11 – 7/31/13 1.9 years
Average	\$1,438,190/year

Barriers

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

Partners

- Michigan State University (MSU)
- Idaho National Lab (INL)
 - **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 of INL.
 - 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



Cellulosic Feedstock Challenges

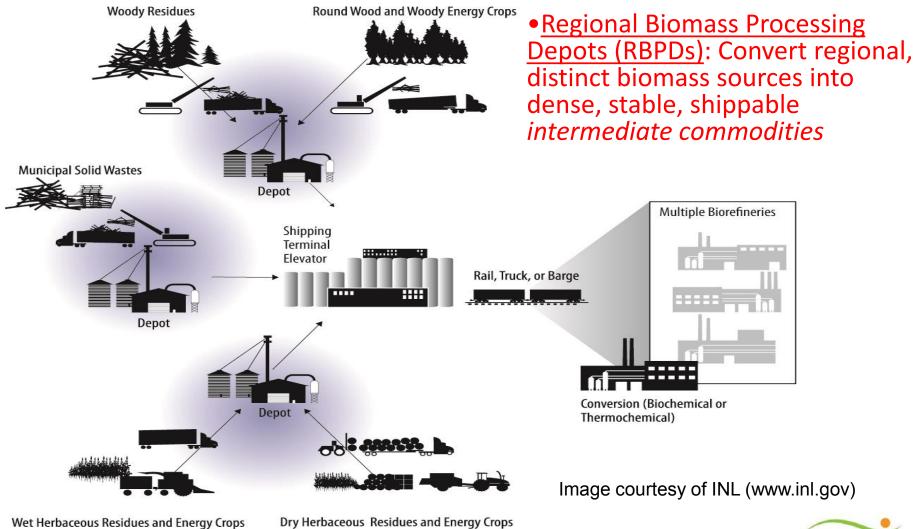
Provide biorefineries with a *robust, viable, supply chain* for biomass feedstocks



Images courtesy of NREL (www.nrel.gov)



Solution: Decentralized Preprocessing and Pretreatment



Regional Biomass Processing Depots

RBPD functions:

- Purchase biomass (corn stover, wheat straw, grasses) from growers
- Short term storage of biomass
- Size reduction of biomass, remove dirt and rocks
- Biomass pretreatment
- Biomass densification

Pretreatment processes for RBPDs must have:

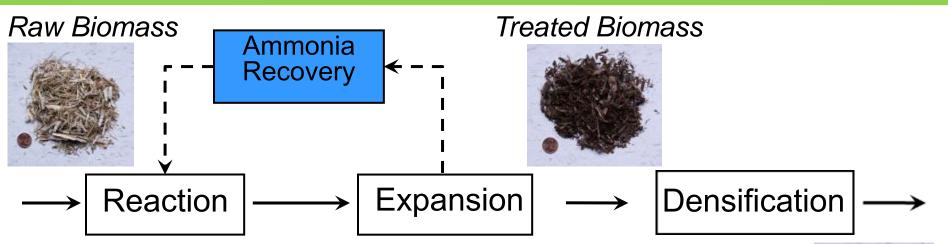
- Low capital cost
- Low water use
- Simple operation
- Suitable to a wide variety of feedstocks and fermentation systems
- Suitable for scaling down (100-200 tons/day)



AFEX Biomass Pretreatment Promising option for RBPDs concept



Ammonia Fiber Expansion (AFEX)



- 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

- AFEX pellets 9-fold denser than biomass
- Stable, storable, readily transportable



AFEX

Pellets



AFEX Is A Distinctive Pretreatment

AFEX Pretreatment advantages for RBPDs:

- Suitable for many types of grasses and agricultural residues
- > No degradation of hemicellulose
- > No wash streams or liquid waste
- Low chemical usage due to ammonia recycle
- Prepares biomass for enzymatic hydrolysis and fermentation
- Improves the digestibility of biomass for use as an animal feed

Can AFEX meet RBPD requirements?

- Low capital cost
- Simple operation
- Easy to scale down
- Stable, conversion-ready product with multiple markets



Pretreatment Challenge

AFEX is one of the most cost-effective pretreatment methods, however, it was still found to be very capital-intensive due to a design based on Pandia-type reactors (AFEX 1) and ammonia recovery system (CAFI and MBI internal).

- Sensitivity analyses show that the primary cost drivers for an AFEX system based on this AFEX1 reactor are:
 - Capital cost
 - Equipment lifespan and maintenance cost
 - Ammonia recovery and recycle not inherent to the system
 - Labor Complex operation requiring skilled operators
 - Energy cost of moving biomass against a pressure gradient

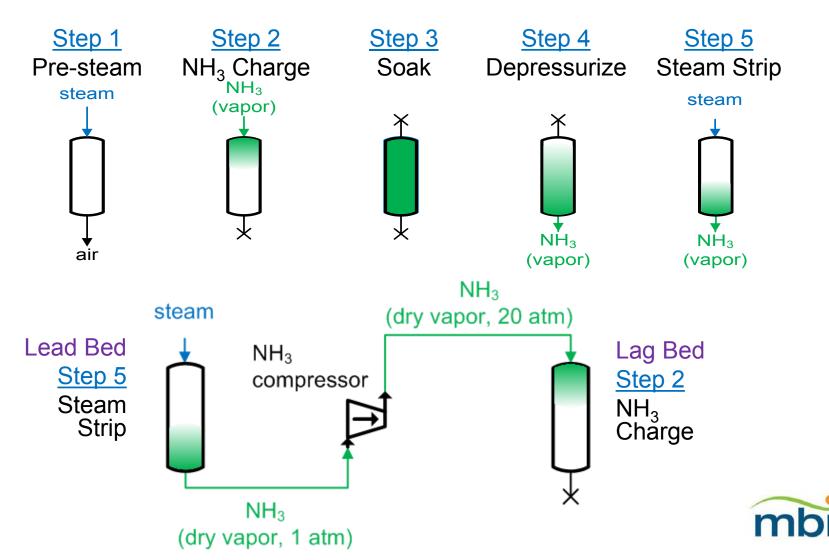
Additionally Pandia-type reactor systems cannot be economically scaled down to a size compatible with RBPDs. This scale-down is a critical factor in RBPDs achieving a feedstock production cost that is commercially viable.



Proposed Solution (AFEX 3)

Concept

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



Early Work: Proof of Concept

Lab scale three-bed skid

fabrication and shakedown:

- Demonstrated NH₃ absorption, desorption, and transfer from bed to bed
- Demonstrated good pretreatment results
 - comparable to stirred batch, with
 - Corn stover
 - Wheat straw
 - Oat hulls
 - Switchgrass





Project Objectives

- Scale up the AFEX 3 system to engineering scale (1 TPD)
- 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



1-Technical Approach

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

B. Preparation of biomass for engineering scale AFEX 3

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

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

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 Go/no Go decision: Will be made in July 2013

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
Go/no Go decision : Intermediate target: 30% reduction in CAPEX and OPEX of AFEX
Will be made in July 2013, and 50% reduction by end of the project July 2014

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

2-Technical Accomplishments Progress/Results



Task A. Address Throughput Risk Using Lab Scale AFEX 3

Bed density has direct effect on the throughput of the process with significant impact on the capital cost of the AFEX 3 system. Bed density of 100 kg/m³ is needed to meet our intermediate cost reduction target (30% compared to the AFEX 1)

Original plan was to use a vacuum blower to load the biomass into the AFEX 3 reactor and to unload the reactor from the bottoms through a fast opening closure.

Target bed density was not achievable with this method

Alternative method, using a cylindrical basket for loading and unloading the biomass into and out of the reactor was developed.

Accomplishment:

- Reached target bed density:100kg/m³ by using the baskets
- Using the basket did not affect the performance and efficacy of the AFEX 3 process



Task A. Address NH3 Recovery Risk

Steam stripping dynamics in a vertical packed bed

Steam density < 0.6 kg/m³

Stripped biomass

Mass transfer zone

Biomass + NH₃

Buoyant effect maintains narrow mass transfer zone –

 NH_3 removed as <u>dry</u> vapor

 NH_3 vapor density > 0.7 kg/m³

Accomplishments:

At aspect ratio of 3: very poor NH_3 recovery At aspect ratios of 6 and 9: acceptable ammonia recovery levels. An aspect ratio of 6 was chosen for the AFEX 3 engineering scale design. Effect of different aspect ratios – 3:1, 6:1 and 9:1 - were evaluated on efficiency of ammonia recovery



Task A. Address NH₃ Recovery Risk

Effect of biomass particle size, type of mill used for particle size reduction, Orientation of the reactor (vertical or horizontal), and bed porosity on the efficiency of ammonia recover was evaluated.

Biomass type	Corn stover				
Mill, particle size	Knife,	Knife,	Knife,	Hammer,	Hammer,
	1/2"	1/2"	1/2"	1"	1/2"
Bed angle (θ _{Bed} , deg.)	90	45	0	0	0
Bed porosity (ε _{Bed} , vol%)	87	87	87	90	85
NH ₃ recovered during steam	28	34	64	91	43
stripping containing >90% ammonia (mass %)					
Residual NH ₃ recovery (mass %)	81	89	97	97	89

Bed porosity calculation:using the bed density ρ_{Bed} and the biomass true density ρ_{True} , as $\epsilon_{Bed} = 100(1 - \rho_{Bed} / \rho_{True})$. Bed angle (θ_{Bed})" refers to the angle between the direction of axial flow through the biomass bed and the local gravity vector.

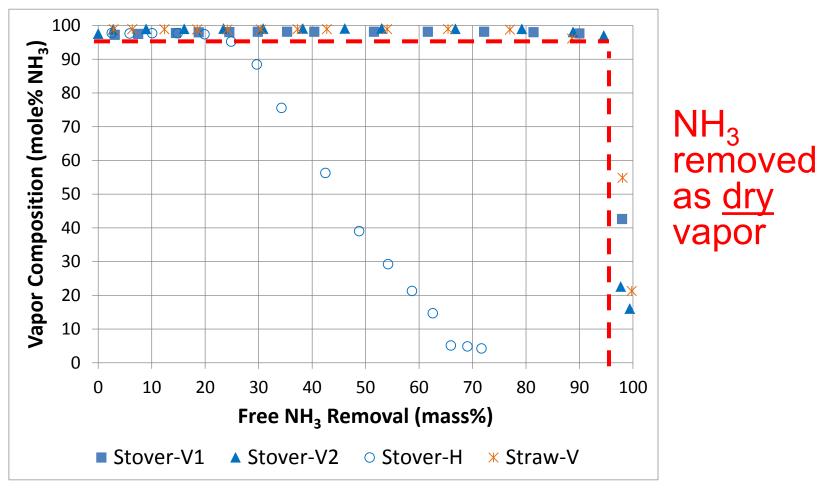
Accomplishments/milestone

Specification for biomass and bed orientation for the engineering scale AFEX 3 system were finalized as highlighted in the above table



Task A. Quality of Recovered Ammonia

Steam stripping dynamics - buoyant effect



Composition of the vapor recovered during steam stripping



Task A. Hydrolysis of Treated Biomass



Task B. Biomass Procurement

This task was led by INL team

Fifty-four large square bales (3-ft x 4-ft x 8-ft) – approximately 30 tons (wet wt.)–of conventional multi-pass, low cob corn stover were harvested and baled by Iowa State University (ISU) on 10/23/2011

Following grain harvest, the stover was winnowed using a Hiniker 5600 Series side discharge winnowing stalk chopper, and baled using a Massey Ferguson MF2170XD large square baler. Several of the bales were cored at harvest and the average moisture and ash contents were determined to be 16.9% and 7.7%, respectively. The average bale weight was 922 lb. The bales were stored under tarps at ISU

INL received the bales on 12/19/2011. Once received, the bales were stacked on pallets and tarped.

Four of these bales were utilized for Task A to provide corn stover with different particle size and particle size distribution.



Task B. Biomass Preparation for Engineering Scale AFEX 3

This task was led by INL team

Corn stover bales were ground to the specification that was determined Under Task A using the single stage grinding method developed for this project Bales above 15 wt% moisture were dried in the PDU.

Per MBI request ground stover were placed into custom plastic-lined bottom-opening supersacks containing 150 +/- 20 lbs worth of material to match their reactor size.

The filled supersacks, numbered 448 sacks were banded three high onto pallets and shipped to WestOne Logistics in Idaho Falls where they were placed in temperature-controlled indoor storage awaiting shipment to MBI

The first shipment of 24 pallets (72 supersacks, roughly 5.4 tons dry weight) were shipped to MBI in mid-December 2012.



Task B. Biomass for Engineering scale AFEX3



Corn stover bales at INL



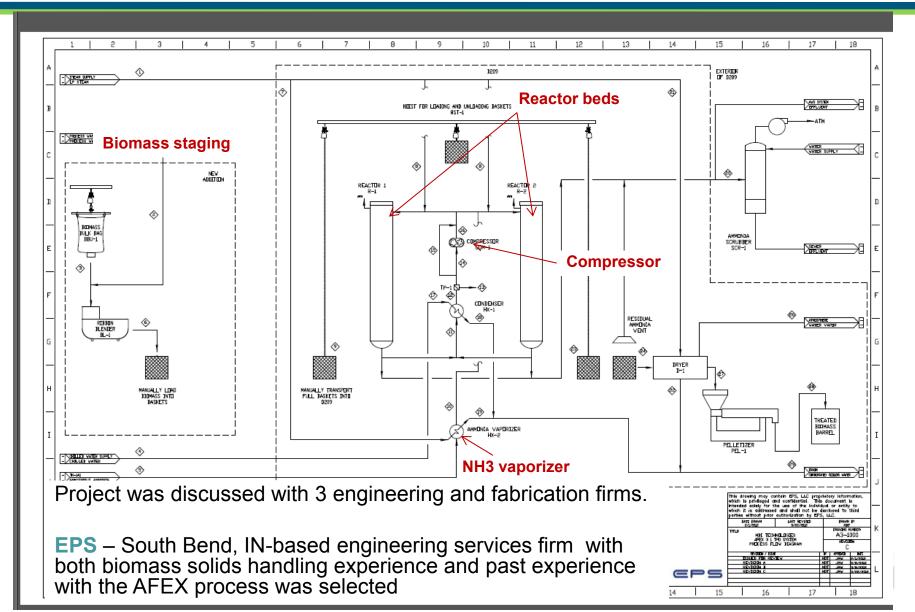
Transferring to the supersacks



Grinding Corn stover at INL







<image/>	Reactor	
	Manufacture	Kennedy Tank and Manufacturing Co, Indianapolis, IN
	Diameter (in)	18
	Height (in)	144
	Material	304 Stainless steel
	Maximum pressure	495 psig@400°F
	Top closure type	Sypris Tube Turns T-Bolt
	Bottom head	Standard weight pipe cap
	Tank shell	1/2" thick rolled and welded



stainless steel plate



Compressor	
Туре	Screw compressor
Model	Frick model RXF 15H
Suction pressure	0 psig
Discharge pressure	300 psig
Flow	3 lb/min





Blender	
Туре	Ribbon blender
Manufacture	Colorado Mill Equipment
Model	RB-2000
Mixing capacity	60 ft ³
Mixer motor	7.5 HP
Agitator RPM	18



Auxiliary Equipment	Purpose
Heat exchanger	Vaporizing ammonia arriving from liquid storage
Condenser	Removing excess water from ammonia stream
Ammonia tank/pump	Provide initial charge of ammonia and makeup ammonia
Scrubber	Removes ammonia vapor from vented fluids
Flow control valves	Allows for control over steam and ammonia inputs
Allen Bradley control system	Simple process control during operation
Ammonia sensor/monitor	Provides alarm in case of ammonia release
Ventilation system	Removes ammonia from atmosphere in case of leak or release
Hoist	Lifts baskets of biomass from floor to reactor
Waste tank	Allows for all drained liquids to be neutralized before being disposed of



Task C. Biomass Unloading Building (BUB)

Supersacks containing biomass received from INL are stored in 2 cargo containers

BUB was constructed on east side of MBI building

Biomass staging is handled in BUB:

- Supersacks are transferred to the building using pallet jack
- Using a hoist supersack is lifted and the biomass is dumped into the ribbon blender
- Moisture of the biomass is adjusted in the blender using the blender built-in misting system
- Moist biomass is dumped into the basket from the large opening at the bottom of the blender
- Using the biomass basket packer biomass is packed in the basket to about 100kg/m³
- Packed baskets are transferred to AFEX3 room (D209) using pallet jacks



Task C. Biomass Preparation







Biomass unloading building



Basket packer







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 40kg of biomass per bed was designed, fabricated and installed



Task D. Shakedown of EngineeringScale AFEX3 System

Method for loading/unloading baskets containing biomass in and out of the reactor beds was developed and successfully tested

Draft for SOPs for biomass preparation and running AFEX3 system were developed

AFEX3 room ventilation system was verified by MSU Environmental Health & Safety (EH&S) office.

Emergency plan in case of ammonia leak and ammonia HAZOP were developed and reviewed by EPS, MBI team and MSU EH&S office

A detailed alarm flow diagram was developed by EPS and reviewed by MBI and MSU (EH&S)

A dry run test was performed by EPS to describe the AFEX3 system to MBI team

Presteaming step was successfully tested

Task D. Shakedown of Engineering Scale AFEX3 System

The system has been pressure tested using N₂

• Leaks were identified that are currently being investigated

The system can not be tested with ammonia until the occupancy permit is issued by City of Lansing.

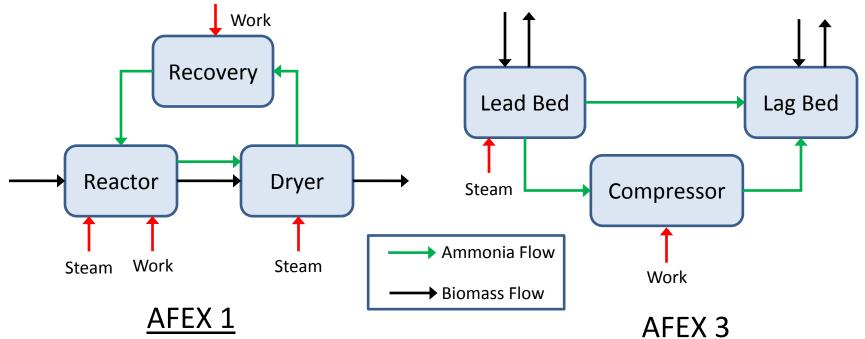
EPS and MBI had several meetings with the Fire Marshal of City of Lansing to finalize the occupancy permit. Required documents have been presented. The permit is expected to be issued by end of April 2013.

Permitting has taken longer than expected and is delaying full shakedown of the system.

We are expecting about 3-4 weeks of delay. However we do not expect a major effect on the overall project timeline



Task E. Comparing AFEX 1 and AFEX 3



- Pandia style horizontal continuous reactor with screw conveyor
- Dryer required with indirect steam heat to recover ammonia
- Two compressors and condensers to separate ammonia and water and concentrate ammonia; >50% of recovered ammonia must be dehydrated

- Vertical packed bed batch reactors in pairs
- Ammonia recovered directly within beds;
- Only one compressor needed, >95% ammonia recovered can be directly reused; < 5% of recovered ammonia must be dehydrated



Task E. Basis for AFEX 1 Design

- No quoted costs available for 100 ton/day Pandia reactor
- MBI has detailed designs for a 50 kg/hr corn stover AFEX reactor and a 1360 kg/ht corn fiber reactor
 - Includes ammonia recovery system
- MBI also has quote for a 1000 ton/day reactor \$14.1MM purchase price
- CAFI project included technoeconomic assessment of 2000 ton/day reactor
- Used as basis to develop scale factor to estimate cost at multiple scales
 - Gradually increased scale factor from 0 to 0.6 as size of reactor increases
 - Obtained similar costs as values quoted to MBI
 - Costs 50% more than CAFI estimate
- Ratio of 54:46 cost of reactor to the ammonia recovery system
 - Similar to CAFI values



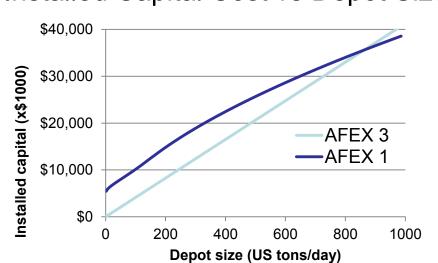
Task E. Basis for AFEX 3 Design

- Based on quoted prices for the engineering scale (~1 ton/day) AFEX 3 reactor and compressor
 - Scale up using 0.6 scaling factor
 - Comparable to estimates of reactor using design equations from Peters, Timmerhaus, and West (2003).
- Assume process improvements in reactor performance
 - 100 kg/m³, 90 minute total residence time
 - One compressor can service 4 reactors



Task E. AFEX 1 vs AFEX 3 Capital Cost

- AFEX 3 significantly cheaper installed capital up to 850 tons/day (~20 million gal EtOH/yr)
- Each component significantly cheaper in AFEX 3
 - Total volume of reactors similar despite high residence time of AFEX 3
 - Dryer cost higher than conventional biomass dryer due to ammonia and not direct contact
- 57% reduction in capital cost

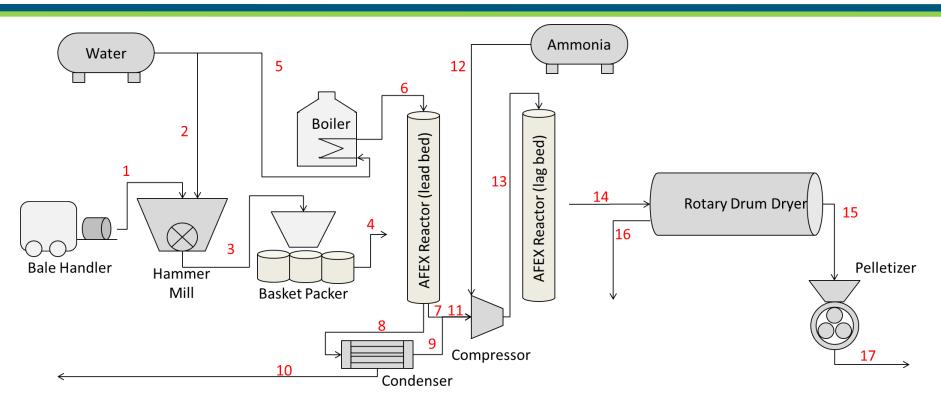


Installed Capital Cost vs Depot Size

Installed Capital Cost for 100 ton/day system

	AFEX 1	AFEX 3
Reactor	\$5,701,000	\$2,688,000
Recovery	\$2,834,000	\$1,920,000
Dryer	\$2,097,000	-
Total	\$10,632,000	\$4,608,000
%Reduction		57%

Task E. Depot



- Auxiliary equipment estimated from IBSAL model (grinder, pelletizer, bale handler, bale storage), obtained quotes (biomass dryer, pellet storage), or design equations (boiler, cooling tower)
- Basket packing for AFEX 3 unknown, internal estimate used

Task E.100 ton/day Depot Capital Cost

	AFEX 1	AFEX 3		
Bale handling	\$104	\$104		
Bale storage	\$15	\$15		
Shredder	\$48	\$48		
Grinder	\$74	\$74		
Ammonia tank	\$90	\$90		
Basket packing	-	\$200		
AFEX	\$10,632	\$4,608		
Biomass Dryer	-	\$900		
Pelletizer	\$216	\$216		
Boiler	\$151	\$81		
Cooling Tower	\$412	-		
Pellet Storage	\$48	\$48		
Eng and Construction	\$3,714	\$1,944		
Total	\$15,503	\$8,315		

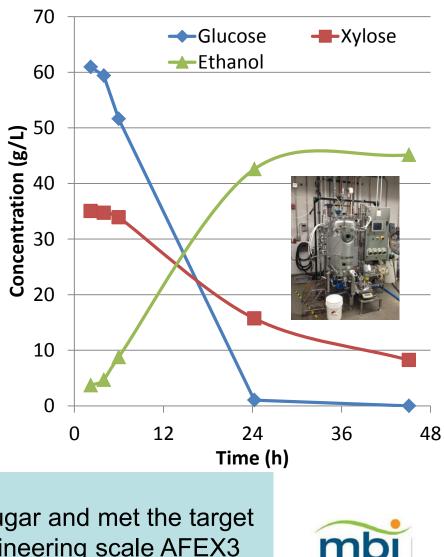
- All numbers are in thousands of dollars
- Only slight differences between designs
 - AFEX 1 requires
 large cooling tower
 - AFEX 3 requires
 basket packing and
 dryer
- 46% reduction in total capital cost

Task F. Testing Fermentability of Sugars from AFEX 3 Treated Biomass

- In stirred tank reactor using corn stover treated in lab scale AFEX3
- SHF fermentation using Z. mobilis (utilizing both C5 and C6)
- Process started with 20% solid loading
- Sterilized water, enzyme, reactor, but not biomass
- Did not remove unhydrolyzed residue prior to inoculation
- Glucose completely consumed within 24 hours
- Xylose ~80% consumed after 48 hours

Accomplishment:

Confirmed the fermentability of generated sugar and met the target will be repeated with biomass treated in engineering scale AFEX3



3 - Relevance

- Strategic Goal: Develop commercially viable technologies for converting biomass feedstocks via biochemical routes
- Overall near term performance goal: decrease cost to \$3 per gallon gasoline equivalent by 2011
- Key technical barriers addressed
 - Bt-E Pretreatment Costs
 - Bt-K Biochemical Conversion Process Integration

Relevance of novel AFEX 3 System

Pretreatment at Regional Biomass Processing Depots allows for improved biomass logistics, ability to produce large scale refineries, and overall simple biochemical conversion process integration

AFEX 3 reduces capital cost of AFEX design by ~50% and operating cost by 30%, allowing for execution at a depot



4 - Critical Success Factors

Critical success factors	Target			
Sufficient throughput: Biomass bed density Cycle time per pair of reactor	Intermediate targetFinal Target100 kg/m3125kg/m3180 min150 min			
Efficacy of AFEX3 engineering scale pretreatment as verified by sugar yields	Sugar yield ≥ conventional AFEX (>75% of available sugars)			
Efficient NH3 recovery and reuse	98% ammonia recovery			
Meet targeted cost reduction of pretreatment	Capital cost reduction Intermediate target Final Target 30% 50%			



5 – Future Work

- Plan through the end of the project.
 - Shakedown of Eng. scale completion
 - Collect Eng. scale mass/energy balance data
 - Final model validation
 - Fermentability of Eng. scale-treated stover demonstrated
- Upcoming key milestones go/no go decision points.
 - Performance milestones for engineering scale
 - Achieving cost reduction target
 - 2nd validation in June, Stage Gate review in July 2013
 - Final validation and Stage Gate in July 2014

 Internal stage gate review with management team and partners will be used to resolve any remaining issues



Gantt Chart for the Remaining Tasks

		20	13			20	14	
Task Name	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
D.1 Shakedown trials of engineering scale AFEX 3 system								
Second on site validation		23-Jun						
First Stage gate		July						
D.2 Operation of the engineering scale AFEX3 system and improving the system								
E. Generate and update techno-economic models of the biomass-to-fuel process.								
F. Determine the quality of pretreated biomass through fermentation use tests								
Last validation and Stage gate							July	



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) Demonstrated ammonia recovery and sugar yields at lab scale
 - 2) Designed, fabricated, and installed engineering scale AFEX 3 reactor
 - 3) Model showing >50% capital cost reduction compared to previous design
 - 4) Demonstrated fermentability of pretreated biomass
- 3) Relevance
 - 1) 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
 - 1) Biomass throughput and ammonia recovery are key critical success factors to be addressed at the engineering scale
- 5) Future Work
 - 1) Complete shakedown and operation of engineering scale reactor
 - 2) Demonstrate efficacy of reactor in terms of throughput, ammonia recovery, and fermentability



Questions?



Publication and Presentation

Campbell TJ, Teymouri F, Bals B, Glassbrook J, Nielson CD, Videto J (2013). A packed bed Ammonia Fiber Expansion reactor system for pretreatment of agricultural residues at regional depots. Biofuels 4: 23-34.

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 AFEXTM-treated corn stover at high solid loadings. Submitted to Biotechnology and Bioengineering.

Campbell T, Teymouri F, Glassbrook J, Senyk D, Bals BD, Nielson CD, Videto JJ, Moore JM. Development of a pilot-scale packed bed Ammonia Fiber Expansion (AFEXTM) process. Presented at 35th Symposium for Biobased Fuels and Chemicals, Portland, OR, May 2, 2013.

Bals BD, Gunawan C, Moore J, Teymouri F, Pardonnet A, Campbell T, Nielson C, Videto J, Dale B. Pelletization and high solids enzymatic hydrolysis of AFEX treated corn stover. Poster presented at 35th Symposium for Biobased Fuels and Chemicals, Portland, OR, April 29, 2013.



Commercialization Efforts

- MBI will license the right to equipment manufacturers to make and sell the AFEX process equipment
 - Due to the simplicity, AFEX 3 system can be manufactured in a factory, transported to a RBPD site for final assembly and installation. This will drive competition which will lead to further advancements in technology

• Efforts : Two major equipment manufacturers have shown interest. Discussion has been initiated with them

- Biorefinery owner need equipment and the rights to run the AFEX process. MBI will license the rights to run the AFEX process directly to biorefinery owners
 Efforts : Several major biofuels and biochemical producers have approached MBI and shown interest. Discussions have been initiated.
- MBI currently is working with major enzyme producers such as Novozyme to develop enzyme for AFEX treated biomass to reach the industry target for hydrolysis yield.
- MBI is actively working on commoditization of AFEX treated biomass and had discussion with farmers throughout the corn belt to engage them in development of RBPD concept