2015 DOE Bioenergy Technologies Office (BETO) Project Peer Review

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

This presentation does not contain any proprietary, confidential, or otherwise restricted information

mbi mission-inspired, market-driven biotech derisking

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 Unites 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

	Total Costs FY 10 –FY 12	FY 13 Costs	FY 14 Costs	FY15 Costs	Total Planned Budget
DOE Funded	$924,626	$1,795,636	$914,919	$41,763	$3,676,944
MBI Cost Share	$544,405	583,340	($19,443)	$3,839	$1,069,254
MSU cost share		$29,922	$50,215	$56,000	$136,137

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

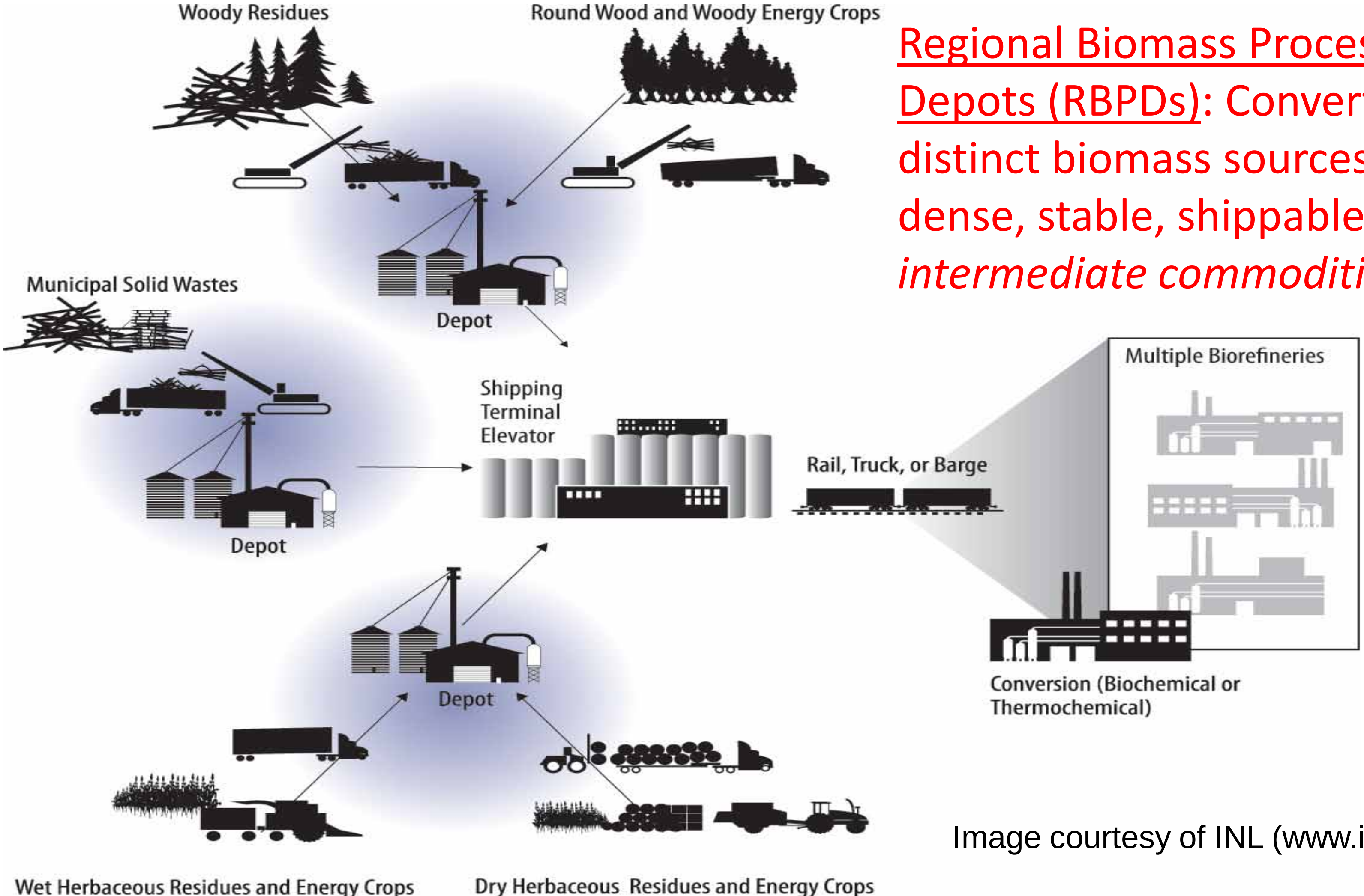
Background - Cellulosic Feedstock Challenges

How can we:

- Upgrade raw biomass to make sugars more accessible?
- Handle, store, transport low-density biomass?
- Establish upgraded biomass as a tradable commodity?

Image courtesy of NREL (www.nrel.gov)

Solution: Decentralized Preprocessing and Pretreatment

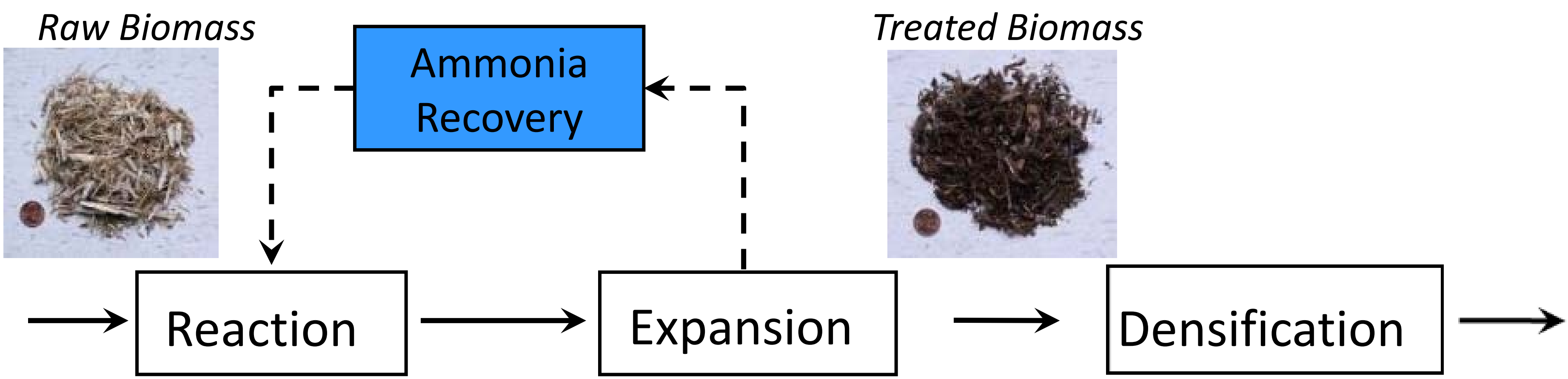


Regional Biomass Processing Depots (RBPDs): Convert regional, distinct biomass sources into dense, stable, shippable *intermediate commodities*

Image courtesy of INL (www.inl.gov)

AFEX Biomass Pretreatment
Promising option for RBPDs concept

Ammonia Fiber Expansion (AFEX)



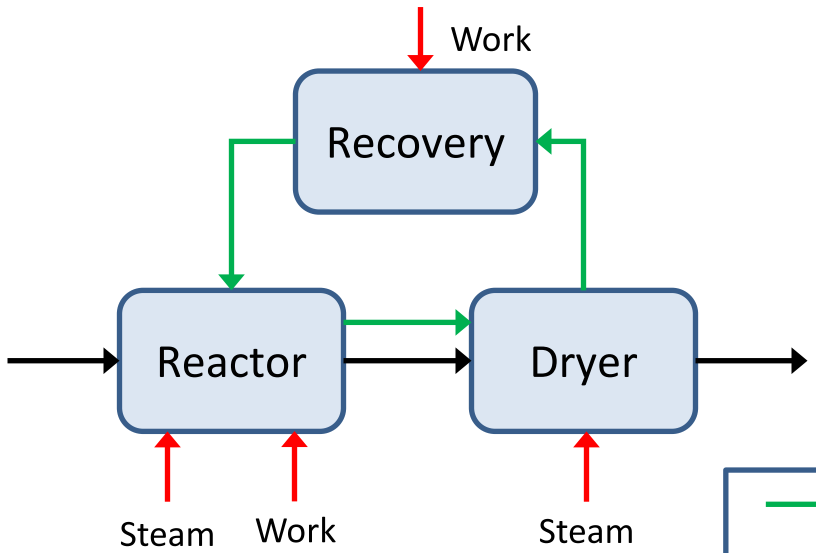
AFEX Pellets

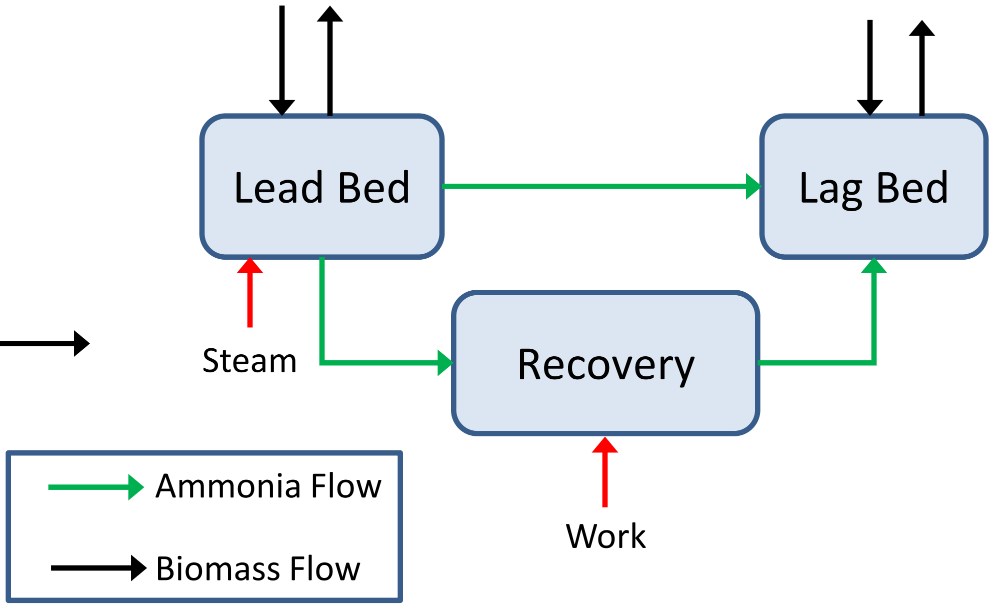
- Moist biomass is contacted with ammonia
- Temperature and pressure are increased
- Contents soak for specified time at temperature and ammonia load
- Pressure is released
- Ammonia is recovered and reused

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

mbi

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

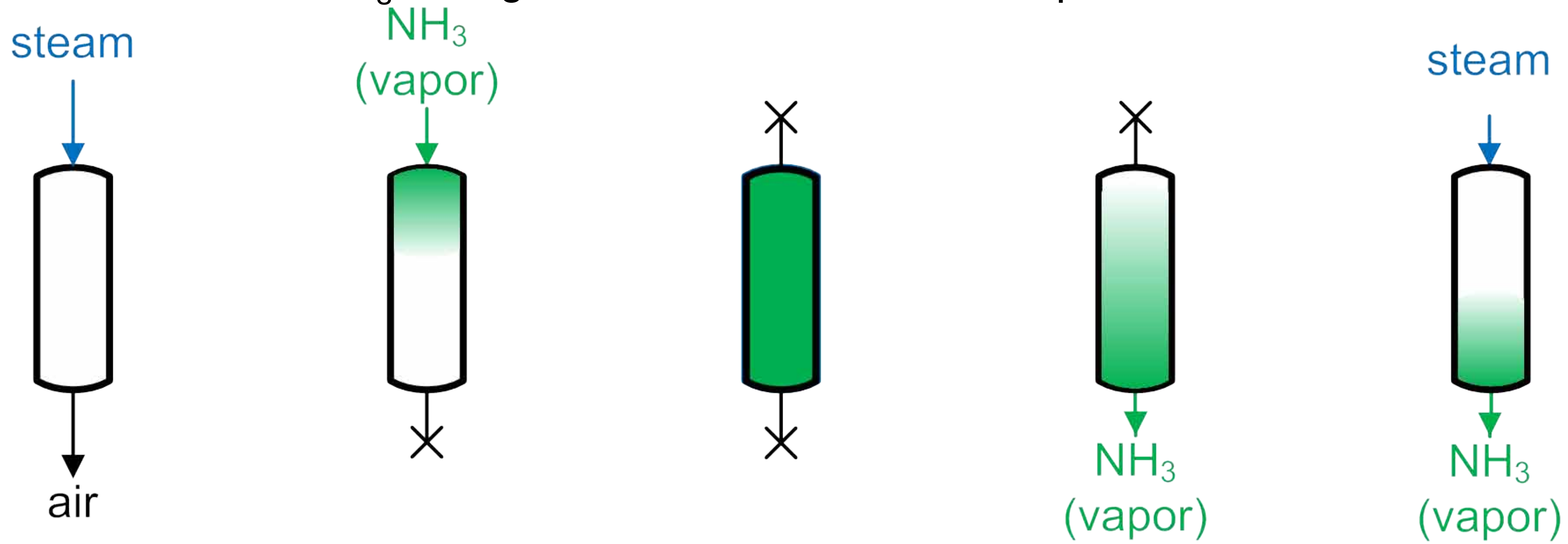
AFEX 3 – Packed Bed AFEX

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:

Step 1	Step 2	Step 3	Step 4	Step 5
Pre-steam	NH_3 Charge	Soak	Depressurize	Steam Strip



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***

mbi

Technical Approach

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

Critical success factors	Target	
Sufficient throughput for commercial-scale reactor (19.5 m^3):	Intermediate target	Final Target
	17 tons/reactor/day	25 tons/r/d
Biomass bed density	80 kg/m3	100 kg/m3
Cycle time per pair of reactor	120 min	110 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%

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-Technical Accomplishments Progress/Results

Progress reported in the last review

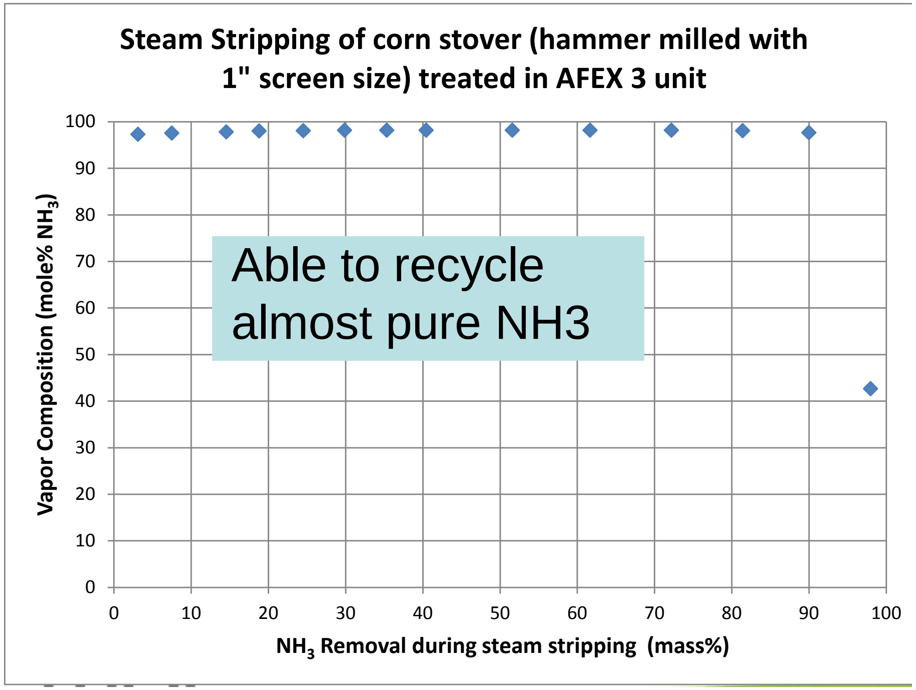
Task A. Addressing Potential Risks Using Lab Scale AFEX 3

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

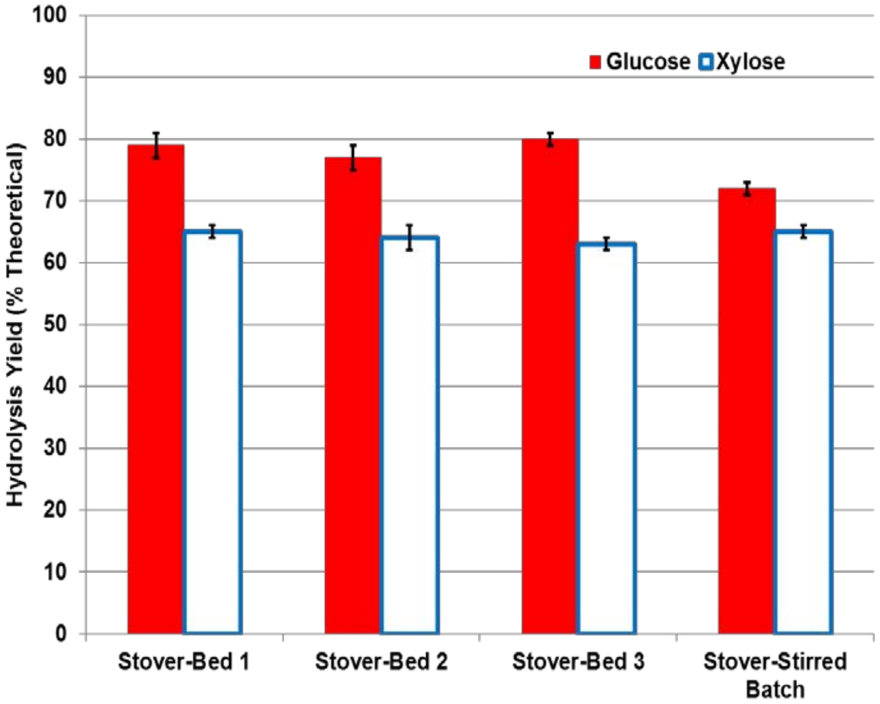
Targets: 95% ammonia recovery and >70% sugar yields at high solid loading

Accomplishments:

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



Composition of the vapor recovered during steam stripping



Task B. Biomass Procurement (INL)

Corn stover bales (25 tons) at INL

Grinding Corn stover at INL

Transferring to the supersacks

Supersacks stored indoors

mbi

Biomass Preparation

Supersacks containing biomass

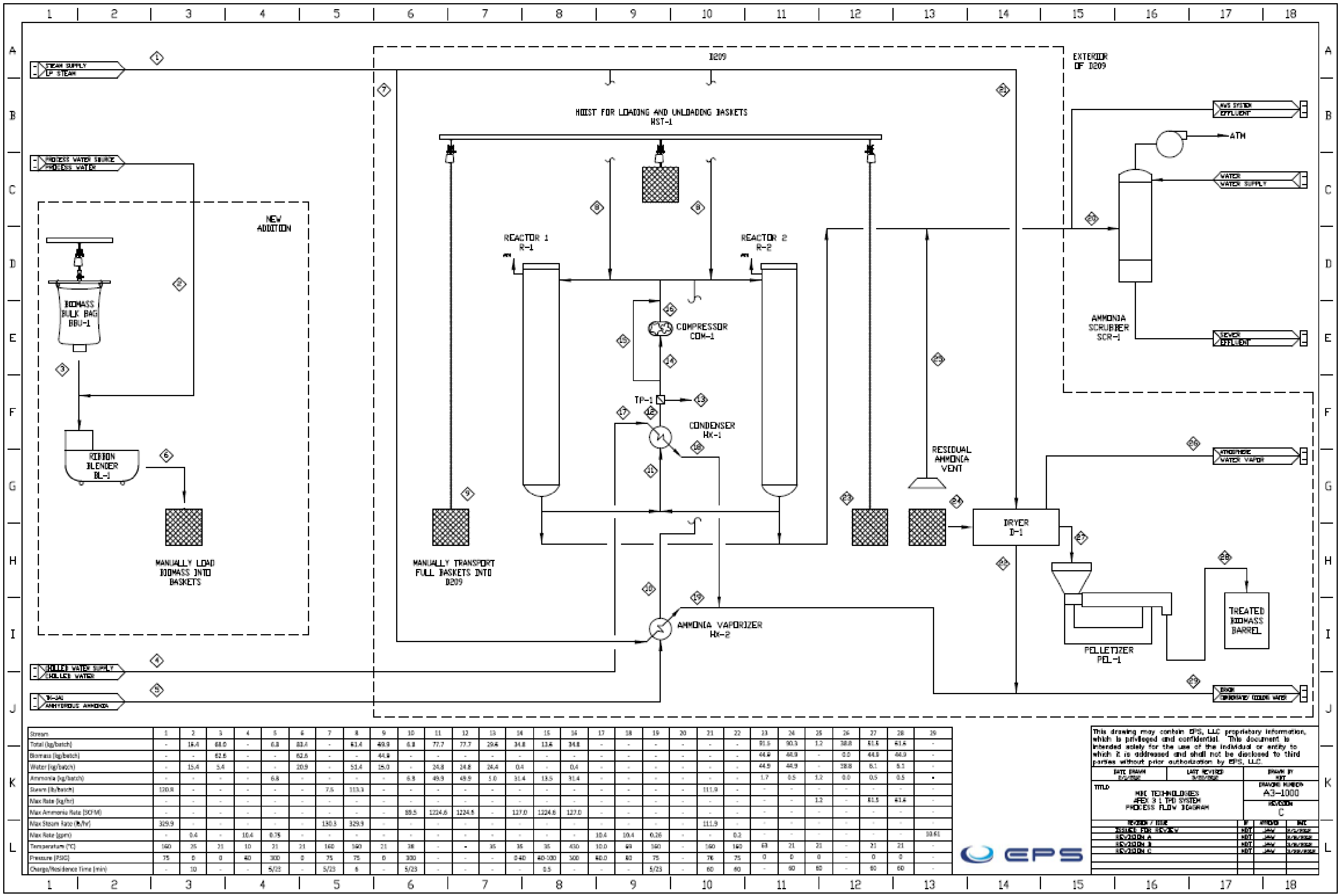
Biomass unloading building

Moisture adjustment in blender

Basket packer

Compressed into baskets, density ~100 kg/m3

Task C. Design of Engineering Scale AFEX 3



Stream	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29
Total (kg/batch)	-	16.4	68.0	-	6.8	83.4	-	61.4	49.9	6.8	77.7	77.7	29.6	34.8	13.6	34.8	-	-	-	-	-	-	91.5	90.3	1.2	38.8	51.5	61.6	-
Biomass (kg/batch)	-	-	62.6	-	-	62.6	-	-	44.8	-	-	-	-	-	-	-	-	-	-	-	-	-	44.9	44.9	-	0.0	44.9	44.9	-
Water (kg/batch)	-	15.4	5.4	-	-	20.9	-	51.4	15.0	-	24.8	24.8	24.4	0.4	-	0.4	-	-	-	-	-	-	44.9	44.9	-	38.8	6.1	6.1	-
Ammonia (kg/batch)	-	-	-	-	6.8	-	-	-	-	6.8	49.9	49.9	5.0	31.4	13.5	31.4	-	-	-	-	-	-	1.7	0.5	1.2	0.0	0.5	0.5	-
Steam (lb/batch)	120.8	-	-	-	-	-	7.5	113.3	-	-	-	-	-	-	-	-	-	-	-	-	111.9	-	-	-	-	-	-	-	-
Max Rate (kg/hr)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.2	-	51.5	61.6	-
Max Ammonia Rate (SCFM)	-	-	-	-	-	-	-	-	-	89.5	1224.6	1224.6	-	127.0	1224.6	127.0	-	-	-	-	-	-	-	-	-	-	-	-	-
Max Steam Rate (lb/hr)	329.9	-	-	-	-	-	130.3	329.9	-	-	-	-	-	-	-	-	-	-	-	-	111.9	-	-	-	-	-	-	-	-
Max Rate (gpm)	-	0.4	-	10.4	0.75	-	-	-	-	-	-	-	-	-	-	-	10.4	10.4	0.26	-	-	0.2	-	-	-	-	-	-	10.61
Temperature (°C)	160	25	21	10	21	21	160	160	21	38	-	-	35	35	35	430	10.0	49	160	-	160	160	63	21	21	-	21	21	-
Pressure (PSIG)	75	0	0	40	300	0	75	75	0	300	-	-	-	0-40	40-100	300	40.0	60	75	-	76	75	0	0	0	-	0	0	-
Charge/Residence Time (min)	-	10	-	-	5/25	-	5/25	6	-	5/23	-	-	-	-	0.5	-	-	-	5/25	-	60	60	-	60	60	-	60	60	-

AFEX Pilot – Valve skid, COMP

Valve skid:

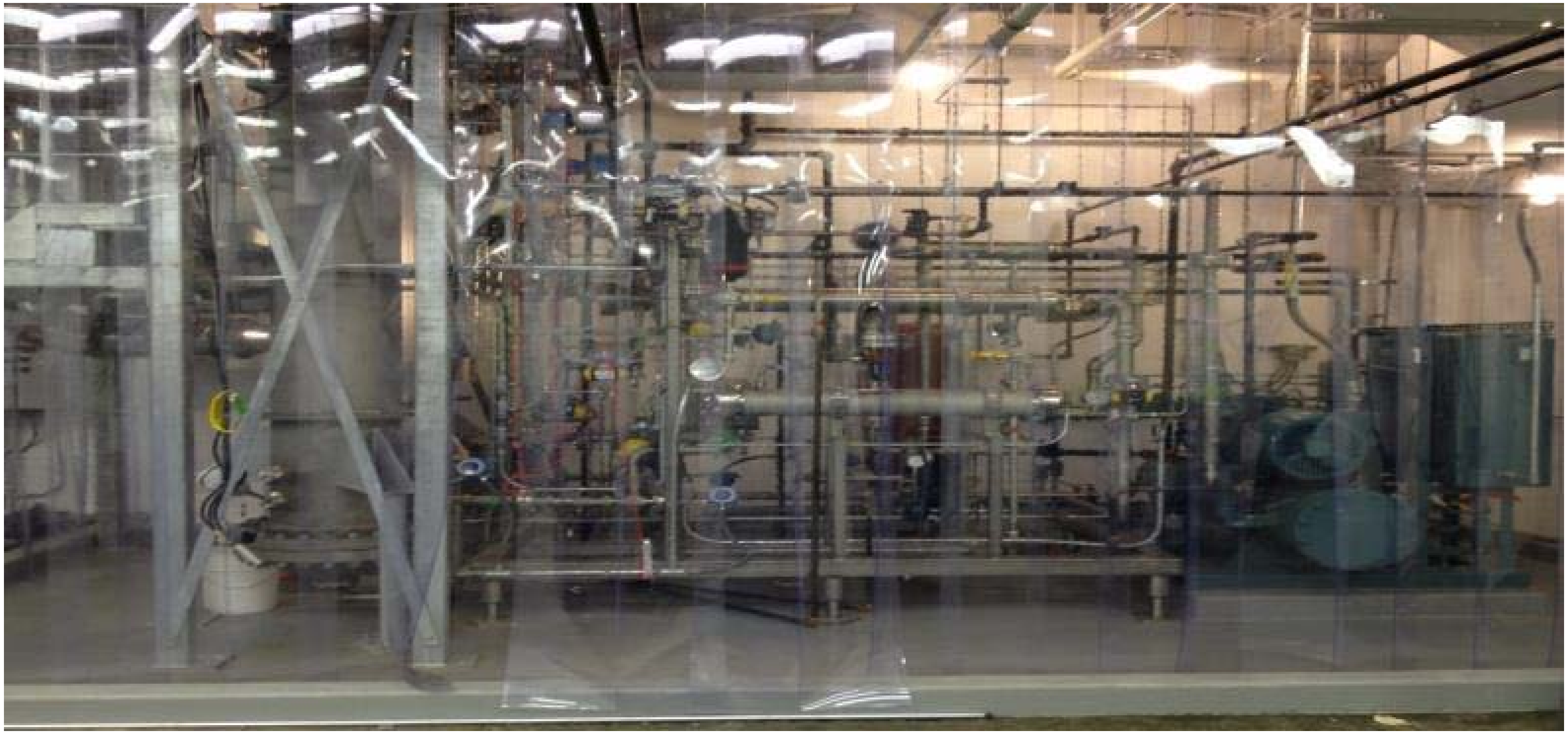
- **Valves –**
  - **60 total, 20 actuated**
- **Instruments –**
  - **12 temp, 12 pressure, 3 flow**
- **Heat exchangers –**
  - **NH_3 Evaporator, condenser**

Compressor:

- **Rotary screw (Frick RXF-15)**
- **Suction 0, discharge 300 psig**
- **NH_3 displacement ≈ 3 kg/min**

Task C. Installation of AFEX3 System

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



Engineering scale AFEX3 system installed in MBI building

Accomplishments/ major milestone:

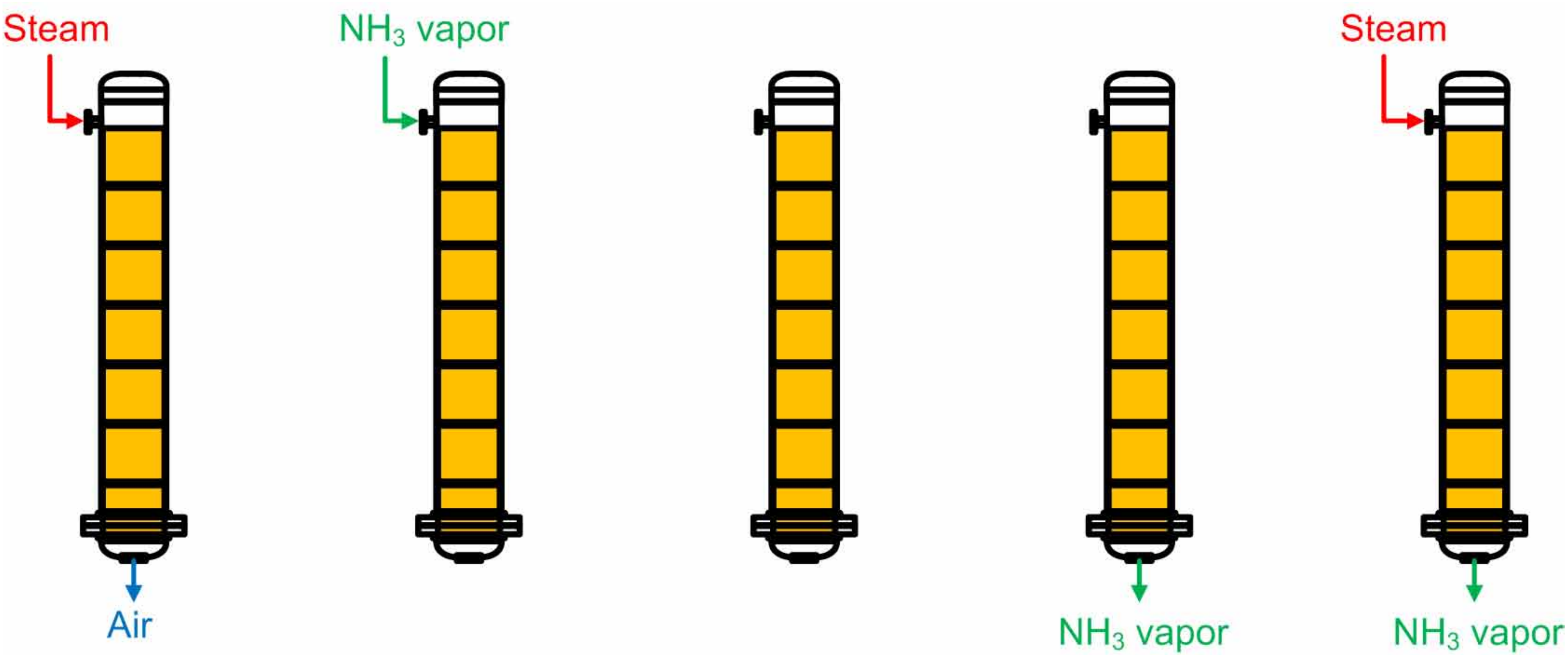
Engineering scale AFEX3 capable of treating more than 35 kg of biomass per bed was designed, fabricated and installed

Progress made since the last review

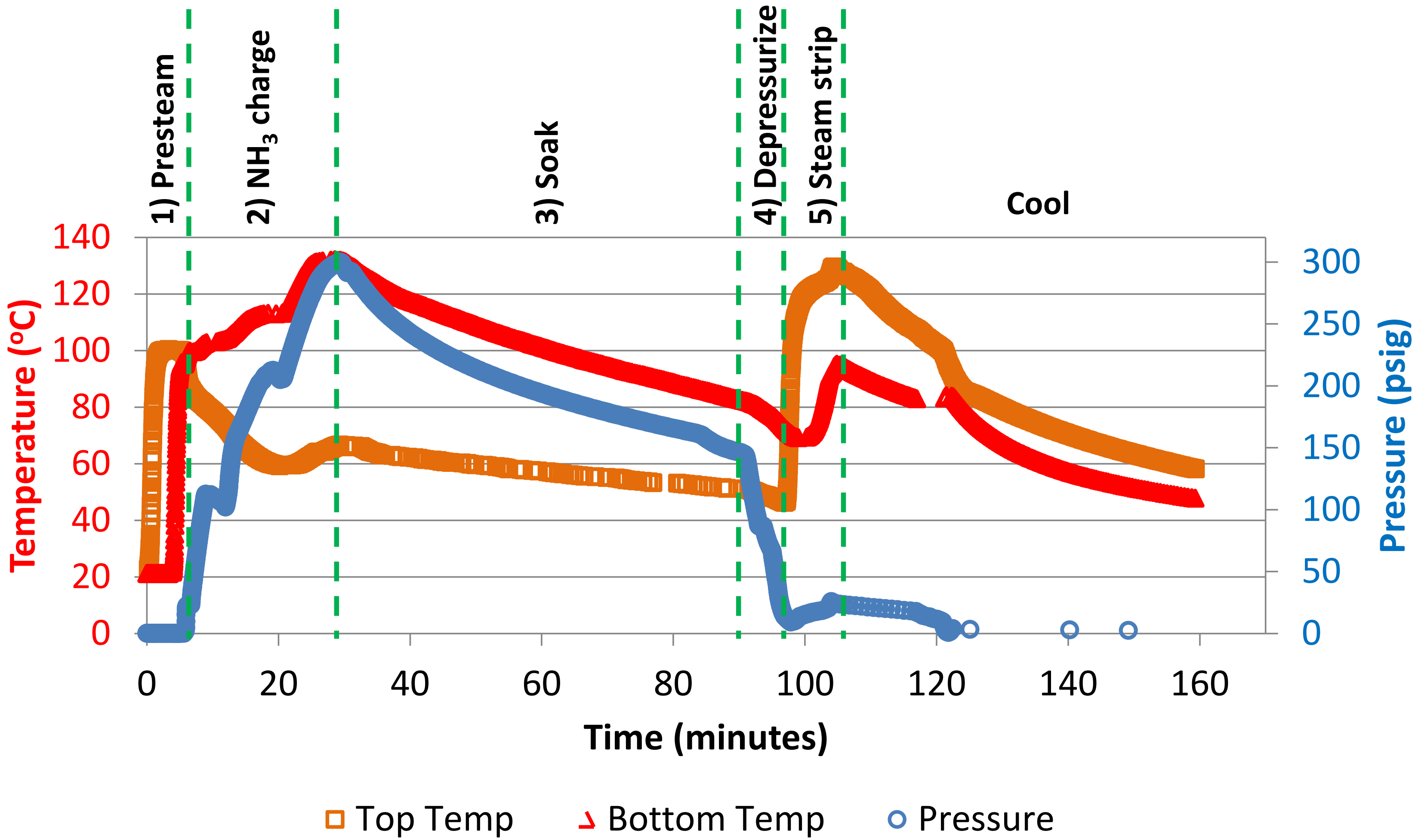
Task D. Operation of AFEX3 pilot scale system

More than 480 beds treated using the five step cycle:

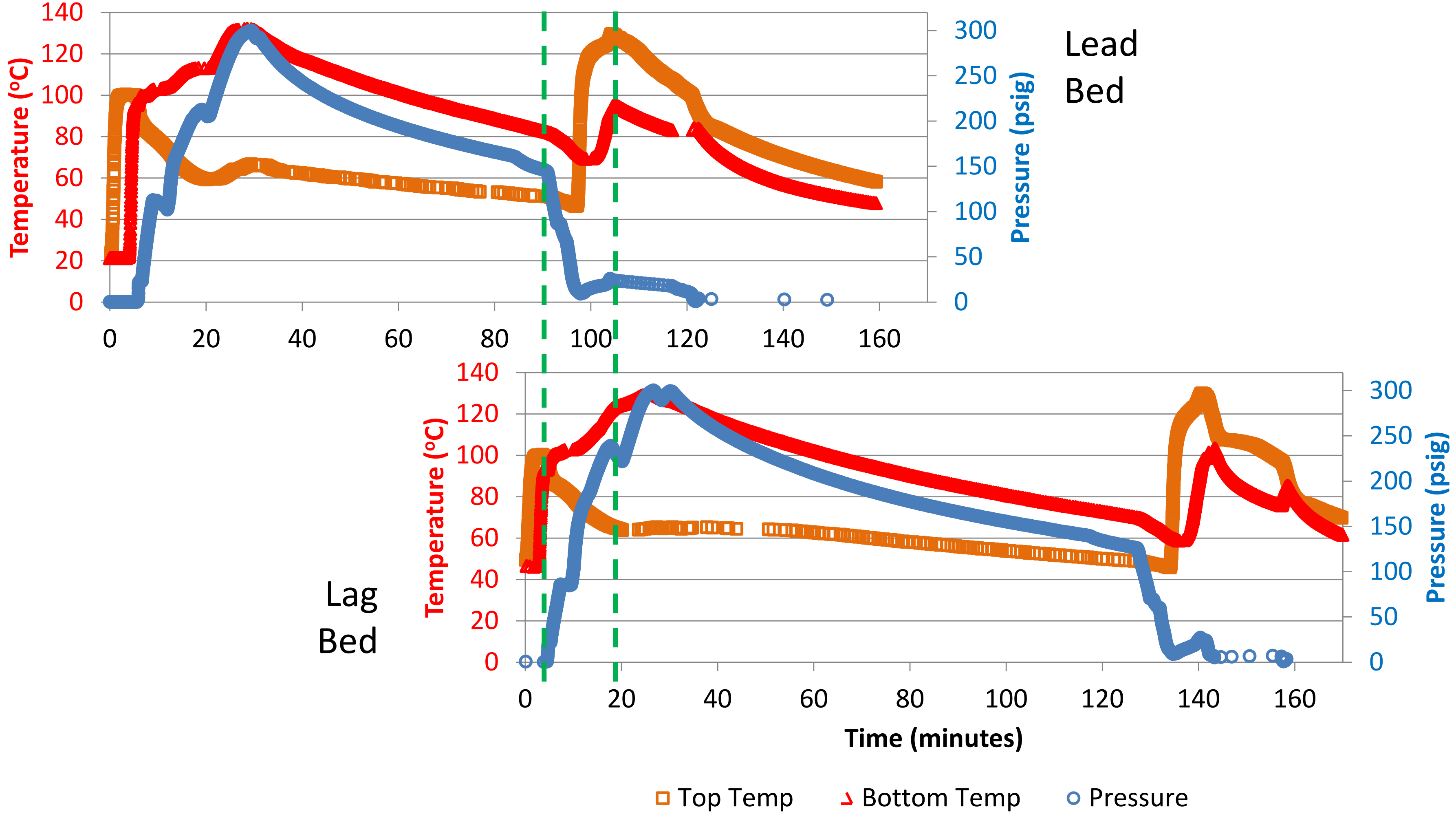
Step 1	Step 2	Step 3	Step 4	Step 5
Pre-steam	NH_3 Charge	Soak	Depressurize	Steam Strip



AFEX Pilot Plant – Treatment Cycle
Bed Temperatures, Pressure

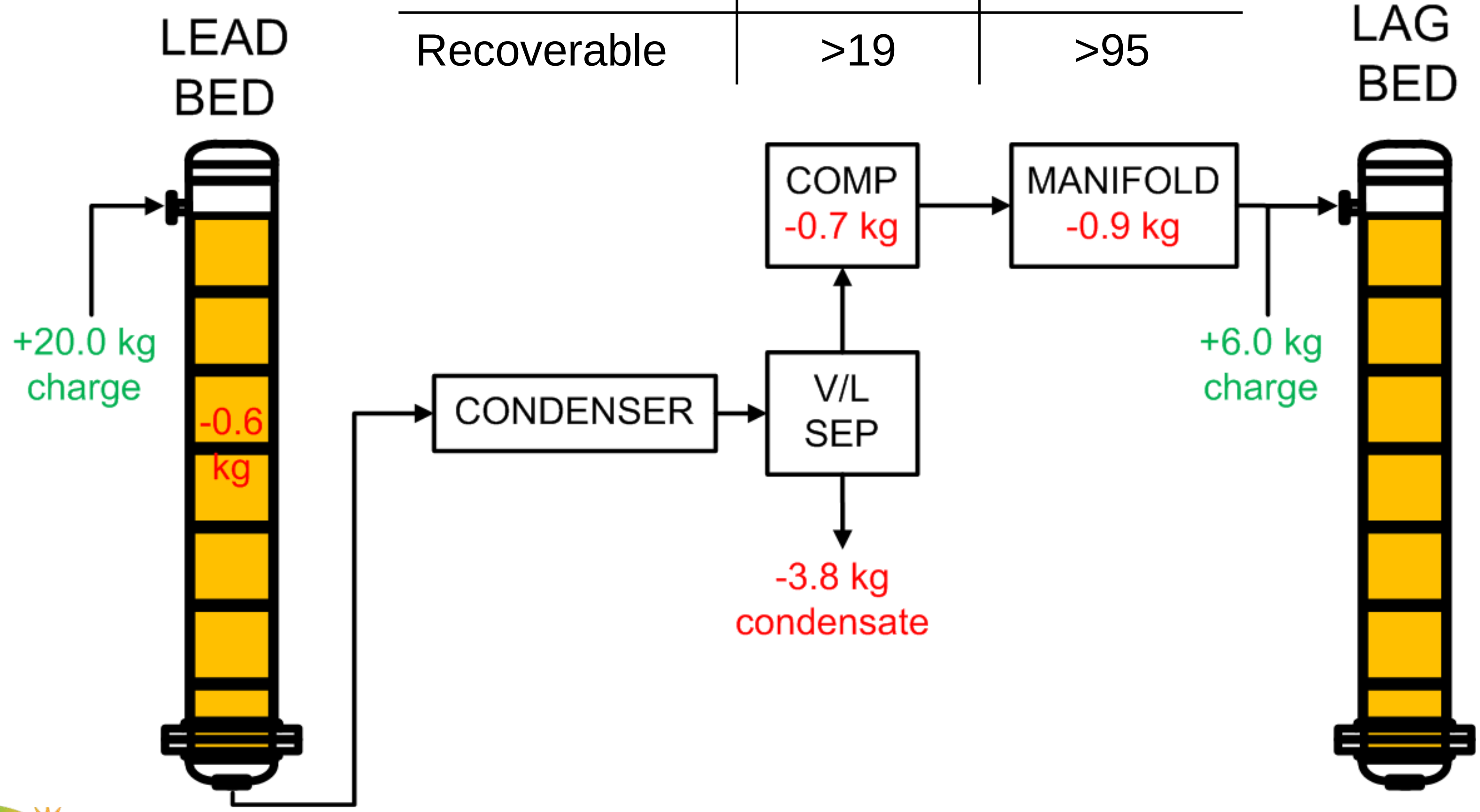


AFEX Pilot Plant – Treatment Cycle
Bed Temperatures, Pressure

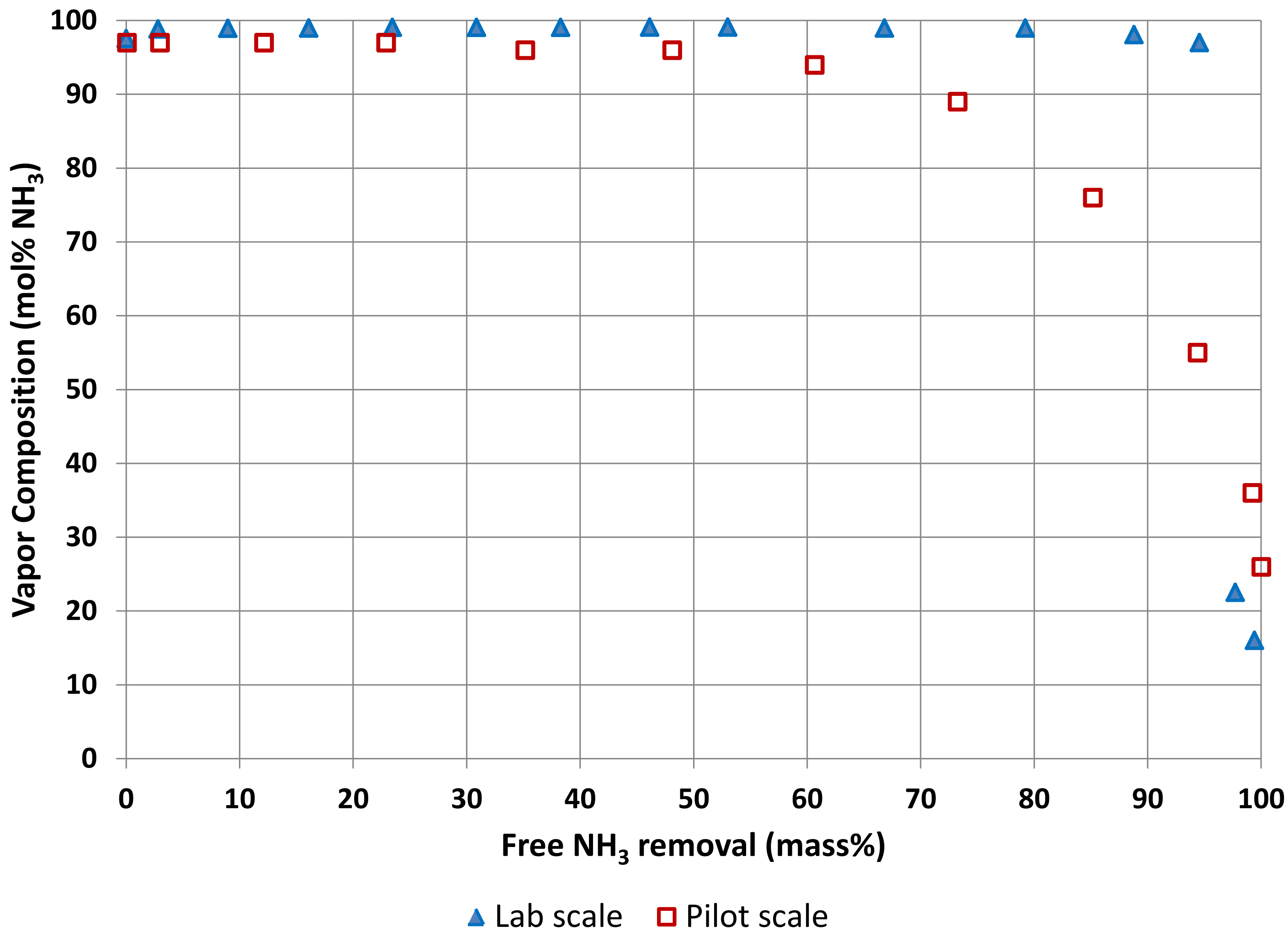


AFEX Pilot Plant – NH_3 Balance (early 2014)

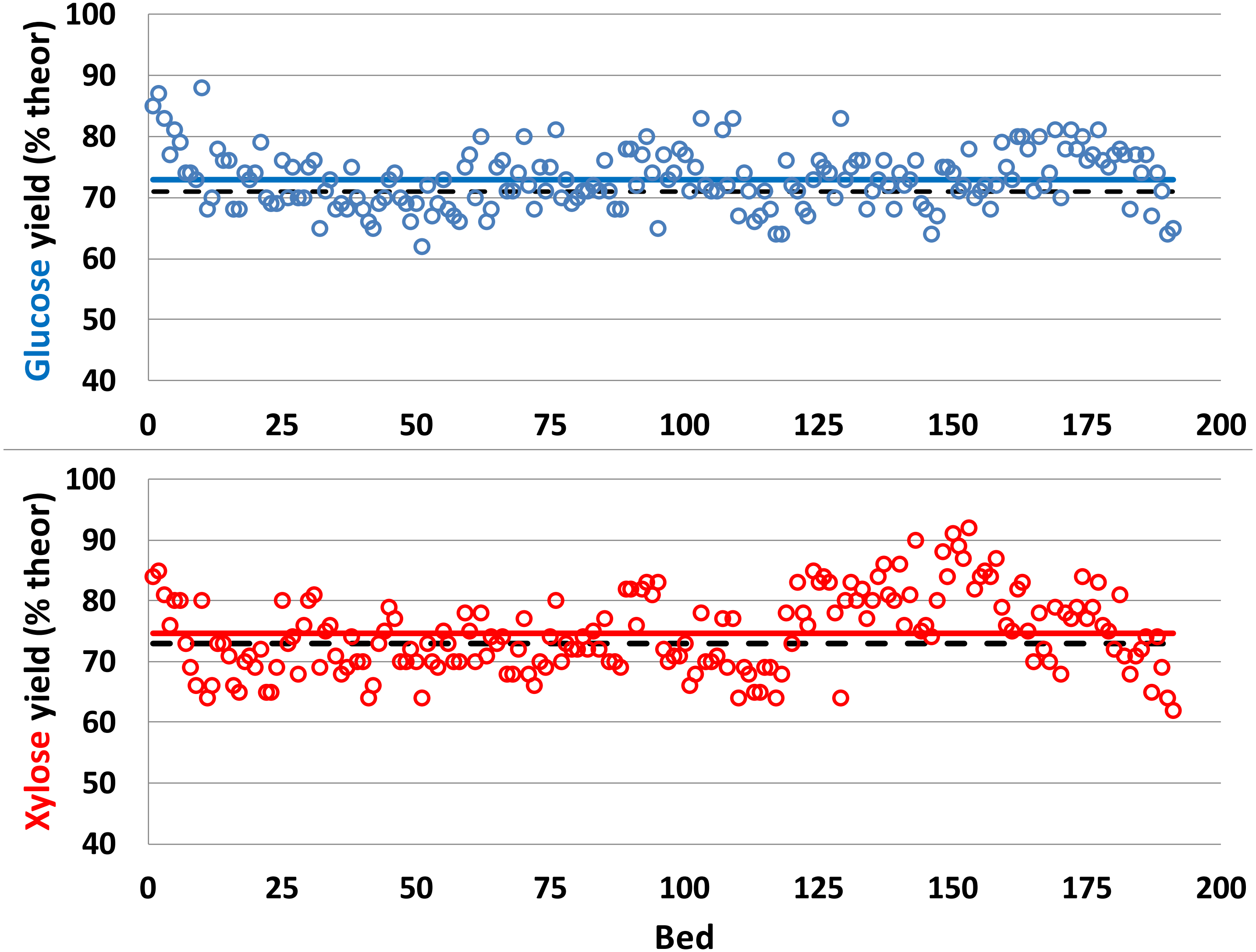
	NH_3 Transfer (kg)	NH_3 Recovery (wt%)
Current	14	70
Recoverable	>19	>95



Steam Stripping Performance
Pilot vs. Lab Scale



AFEX-Treated Corn Stover Enzyme Hydrolysis Results



Solid line: Average of all the runs

Dashed line: Benchmark performance

AFEX 3 Pilot Plant - Compressor Issues

May 2014:

- Shaft seal leak
- Bearing cages disintegrated
- Heavy corrosion and scaling throughout machine
- Cause: H_2O accumulation

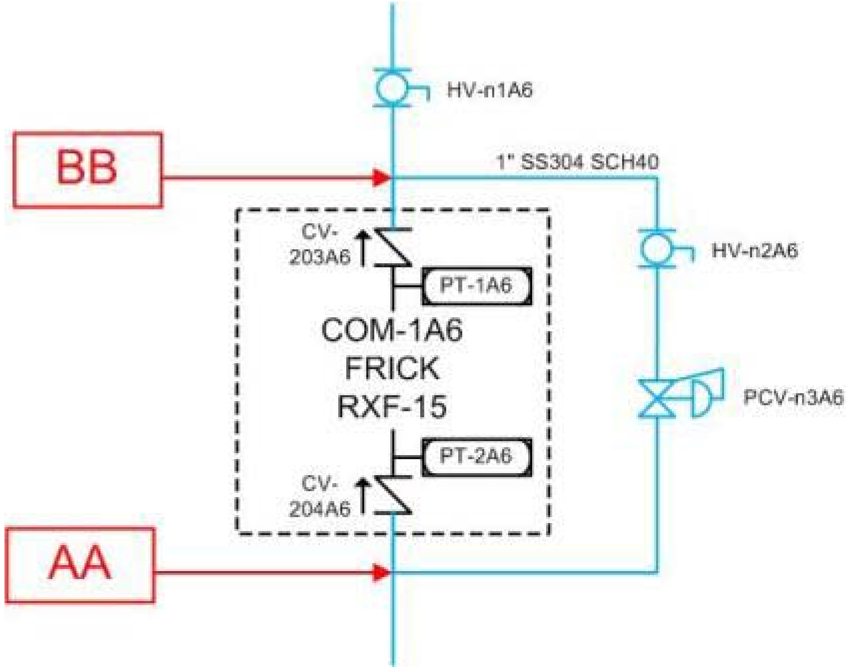
AFEX 3 Pilot Plant - Compressor Issues

Issue	Causes	Problems	Solutions
H_2O accumulation	• Stop/start duty • Mist formation in condenser	• Corrosion • Oil degradation	• Hot gas bypass • Mist eliminator
Overheating	Control valve malfunction	Load inhibition at high discharge T	Proper control valve settings
Compressor wrong approach for AFEX?	Compressor control incompatible with batch process?	• Continued H_2O accumulation • Others?	Develop absorption approach

AFEX Bed-to-Bed NH3 Transfer

Hot Gas Bypass line installed Oct 2014

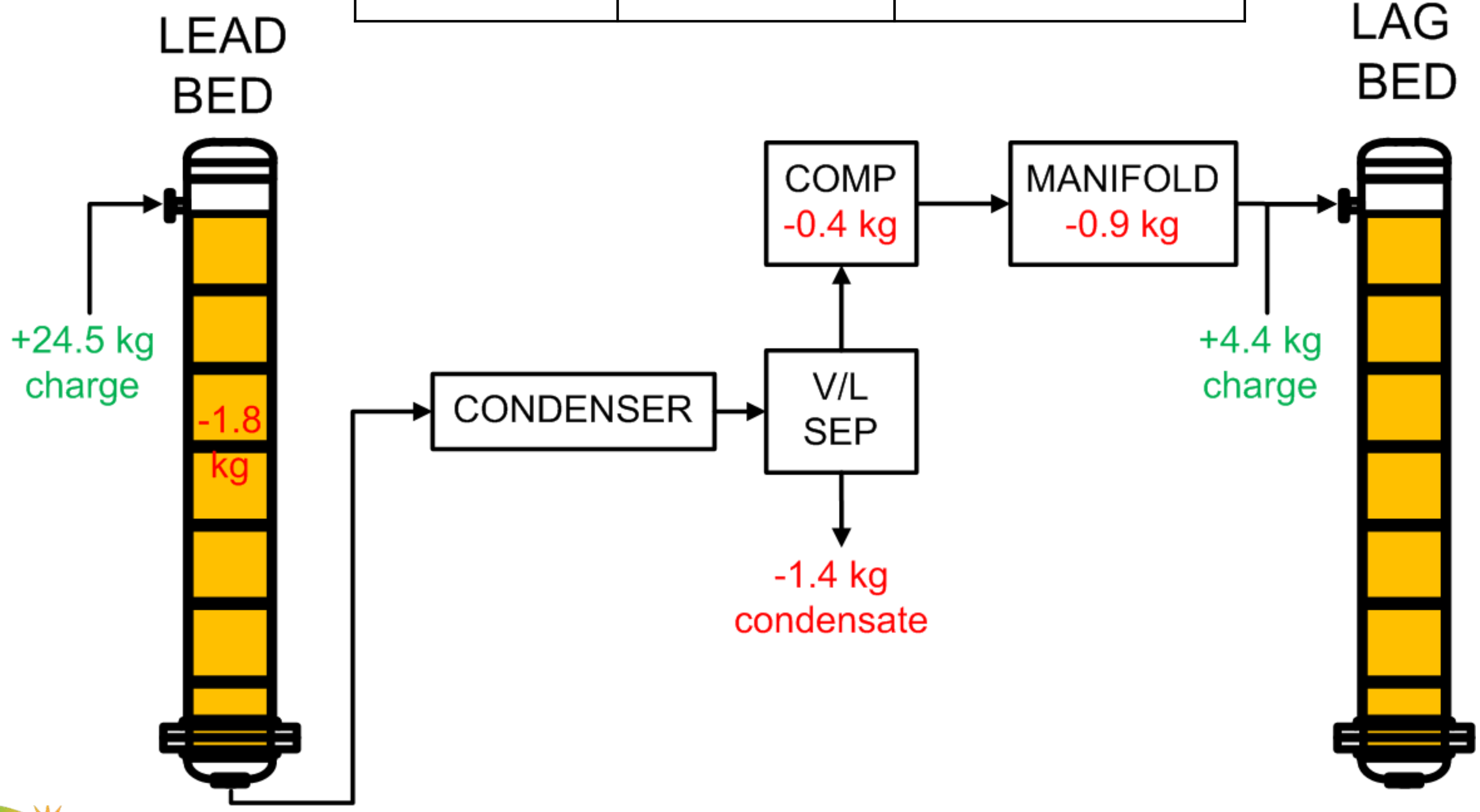
- Allows continuous operation between bed-to-bed NH_3 transfers
- Compressor maintains operating temp 220ºF
- Oil P drop problem
- High electrical load



•Investigating alternative approach for ammonia recovery – remains ongoing

AFEX Pilot Plant – Current NH3 Balance

	NH_3 Transfer (kg)	NH_3 Recovery (wt%)
Validation Run	20	82
Recoverable	23	94



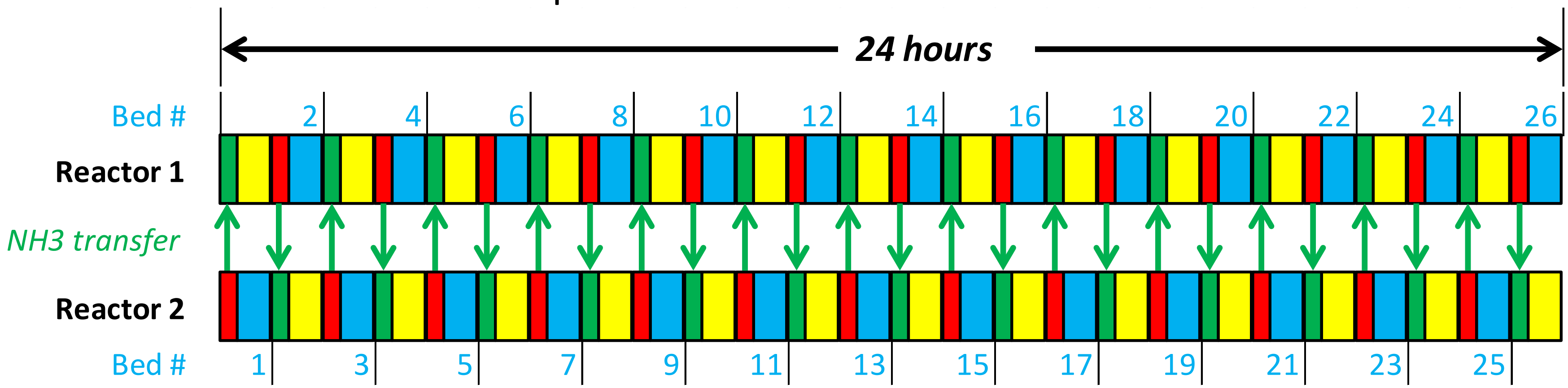
AFEX Treatment Cycle

Single Reactor: cycle time based on pilot plant results

Steps	NH_3 Charge	Makeup NH_3 Add → Soak	DePressurize → Steam Strip	Unload → Reload → PreSteam
Time (minutes)	19	36	19	36

Reactor Cycle Time = 110 minutes

Reactor Pair: 24 hours of operation



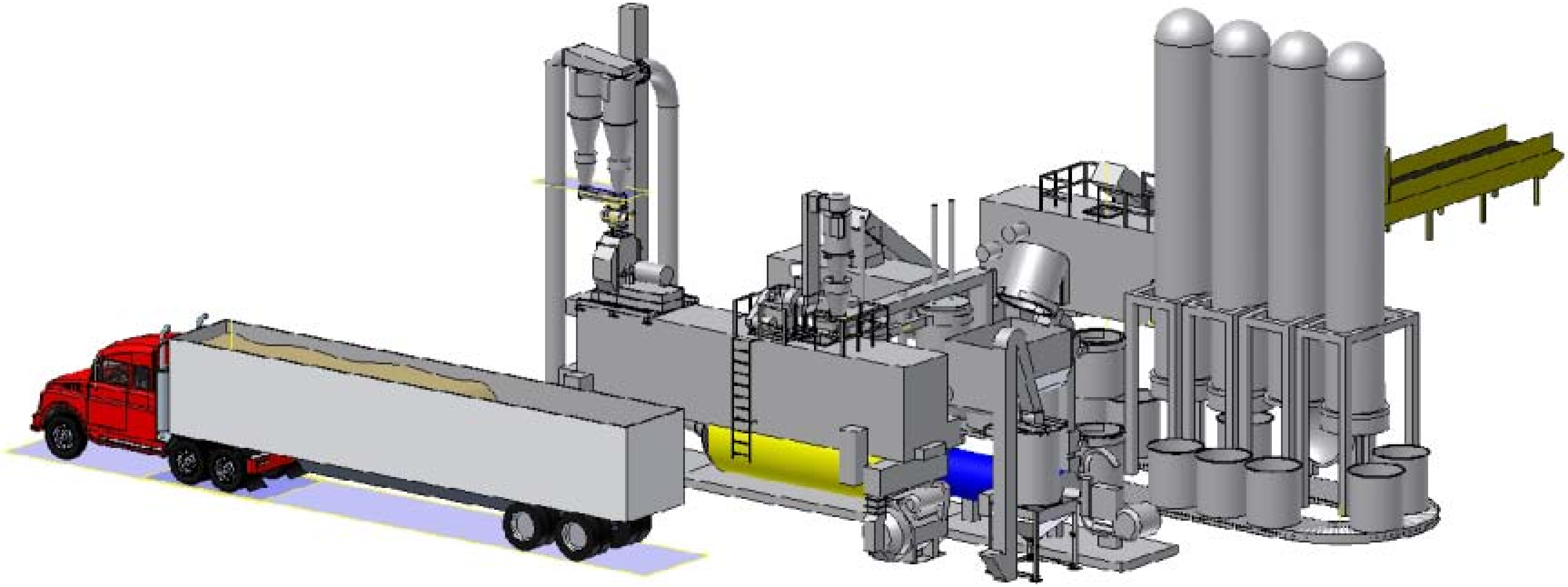
Depot-Scale Reactor Pair Output: V_{Bed} = 19.4 m^3 ; ρ_{Bed} = 100 kg(dry)/m^3

1,940 kg dry biomass / Bed
26 Beds / Pair / Day
} = 50 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**

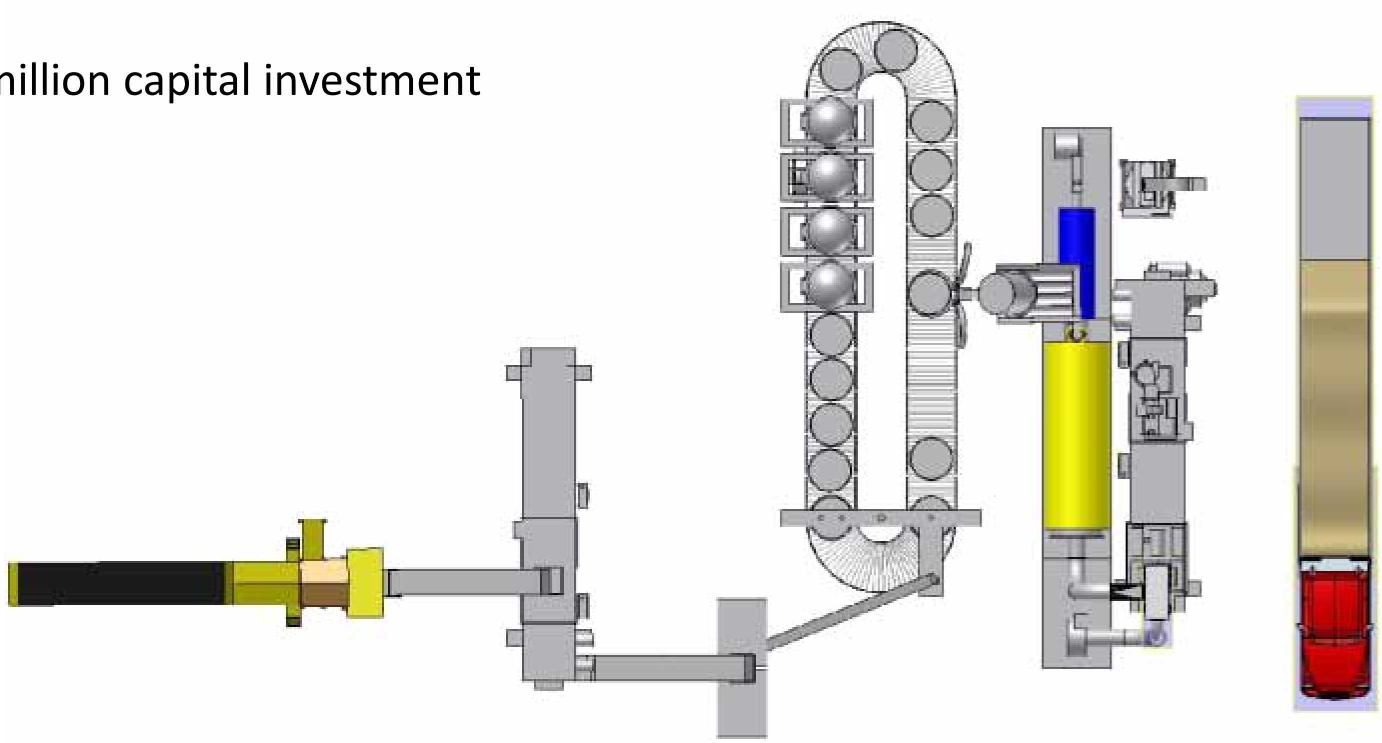


100 TPD AFEX depot CAD drawing by Corrie Nichol, INL

mbi

AFEX Depot

$9.5 million capital investment



100 TPD AFEX depot top view drawing by Corrie Nichol, INL

Depot cost analysis

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

Total cost of pellets is ~$160/DMT

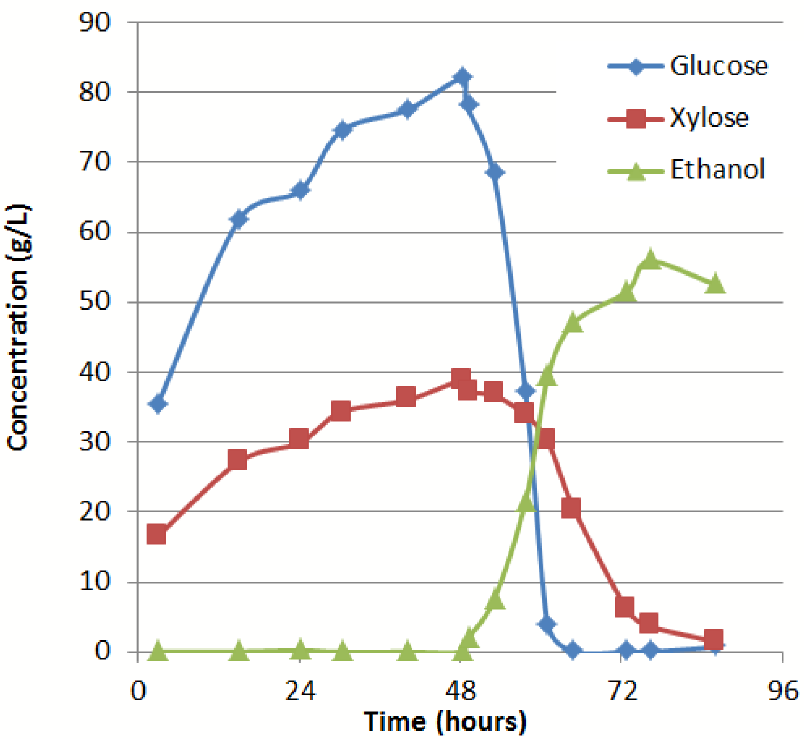
Cost determined by:

- 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

4 - Relevance

DOE multi-year plan performance goal (2013):

Through R&D, make cellulosic biofuels competitive with petroleum-based fuels at a modeled cost for mature technology of $3 per gallon of gasoline equivalent (GGE) ($2011) based on EIA projected wholesale prices in 2017. Help create an environment conducive to maximizing the production and use of biofuels by 2022.

- **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**

mbi

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

Critical success factors	Target	Actual
Reactor Throughput	25 tons/reactor/day	25 tons/reactor/day
Sugar yields	>75% of available sugars	75% at pilot
Ammonia Recovery	98% ammonia recovery	94% ammonia recovery
Capital Reduction	50%	46%

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.

Campbell T, Teymouri F, Glassbrook J, Senyk D, Bals BD, Nielson CD, Videto JJ, Moore JM. Pilot-Scale De-Risking of AFEX Performance and Applications. Presented at 36th Symposium for Biobased Fuels and Chemicals, Clearwater, FL April 30, 2014.

Amber N. Hoover, Jaya Shankar Tumuluru, Farzaneh Teymouri, Janette Moore, Garold Gresham. Effect of pelleting process variables on physical properties and sugar yields of ammonia fiber expansion pretreated corn stover. Bioresource Technology 164 (2014) 128–135

mbi

Publication and Presentation

Ian J. Bonner, David N. Thompson, Farzaneh Teymouri, Timothy Campbell, Bryan Bals, Jaya Shankar Tumuluru. Impact of Sequential Ammonia Fiber Expansion (AFEX) Pretreatment and Pelletization on the Moisture Sorption Properties of Corn Stover. Manuscript submitted to Drying Technology

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.

mbi