

# Developing a Biomass Industry in North Dakota

A Report Submitted to  
The North Dakota Industrial Commission  
Biomass Incentives and Research Program  
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## Executive Summary

A consortium led by NDSU is currently engaged in a project to develop and commercialize technologies to produce fuels and materials from biomass feedstock in North Dakota. The first major milestone in the commercialization effort is to address key economic and engineering questions to determine the technical and economic feasibility of a pilot scale production process. This study, supported by the Biomass Research and Incentive Program of the North Dakota Industrial Commission was undertaken to address those questions. This, the *detailed investigation*, has three objectives: 1) complete a front end engineering and design (FEED) study for a pilot scale plant, 2) determine the best extraction methods for refining cellulosic nanofibers and definition of the process, 3) prepare a strategic business plan for the integration of public and private sector resources to provide investment for pilot plant construction.

Objective 1: The FEED study completed during the detailed investigation phase addressed key engineering and economic questions to quantify the technical and economic feasibility of a pilot scale production process for an integrated biorefinery using AFEX pretreatment. A preliminary design for an AFEX and CAFEX (continuous AFEX) pretreatment has been completed (Appendix A). AFEX processing conditions and enzyme hydrolysis conditions were refined, and organisms capable of utilizing both 5 and 6 carbon sugars were screened.

Objective 2: Microfibrillated cellulose (MFC) appears to be more promising than cellulose nanofibers (CNF). MFC have similar properties but can be produced using a simpler process that costs less than producing CNF. Additional research is needed in this area. Good dispersion of the fibers is critical to producing a polymer with desirable mechanical properties. While preliminary studies appeared to have solved the dispersion issue, these results have not been replicated.

Objective 3: Findings from the detailed investigation were used to develop a proposal for the U.S. Department of Energy for the construction of a fully integrated biorefinery pilot plant using AFEX pre-treated biomass. Funding would have moved the project from the *detailed investigation* to the *development phase*; however the project was not funded due to questions regarding ammonia recovery. Ammonia recovery is a key economic parameter that will drive the feasibility of an integrated biorefinery using AFEX pretreated biomass that will have to be demonstrated before moving to the development phase and construction of a pilot scale plant. Because the overall business climate is more risk averse than at the onset of this project, without further de-risking investors are not willing to make the significant investment necessary to build a pilot plant. Work has begun on an alternate means to demonstrate ammonia recovery at a cost far less than that of an integrated pilot plant. The *development phase* will likely take place in two stages. The first will be to demonstrate ammonia recovery using the new approach currently being developed and the second would be to identify and secure a private sector partner(s) for the construction of a pilot scale plant.

Capitalization and financial performance were estimated assuming ammonia recovery. A commercial plant selling ethanol for \$2.19 per gallon yields a 21.6 percent return on investment. Total investment costs were estimated to be \$379 million. Life cycle analysis for an integrated biorefinery indicates a 65 percent reduction in greenhouse gas emissions and a 73 percent reduction in fossil fuel usage. Bench scale testing has demonstrated 94 percent of theoretical yield of fermentable sugars, 98 percent recovery of ammonia and 5 percent ethanol titer in high-solids enzymatic hydrolysis and fermentation AFEX pre-treated corn stover. This data demonstrates that an AFEX integrated biorefinery has significant commercial potential.

## INTRODUCTION

The economic potential of bio-based fuels and materials is substantial. A consortium led by NDSU is currently engaged in a project to develop and commercialize technologies to produce fuels and materials from biomass feedstock in North Dakota. The first major milestone in the commercialization effort is to address key economic and engineering questions to determine the technical and economic feasibility of a pilot scale production process. This study, supported by the Biomass Research and Incentive Program of the North Dakota Industrial Commission was undertaken to address those questions. This, the *detailed investigation*, has three objectives: 1) complete a front end engineering and design (FEED) study for a pilot scale plant to demonstrate the commercial potential of producing fuels and materials from biomass in North Dakota, 2) determine the best extraction methods for refining cellulosic nanofibers and define the process, 3) preparation of a strategic business plan for the integration of public and private sector resources to provide investment for pilot plant construction. The strategic business plan will discuss the potential nature of operations as well as examine potential markets, capitalization requirements and projected financial performance. Findings for Objective 3 are discussed in the following sections. Findings from Objectives 1 and 2 are detailed in Appendix A, “Developing a Biomaterials Industry in North Dakota” and Appendix B, “Cellulose Nanowhisker Technology Development: Investigation into Utilization of Wheat Straw Residue from Ethanol Production (AFEX + SSF).

## STRATEGIC BUSINESS PLAN

The FEED study completed during the detailed investigation phase addressed key engineering and economic questions to quantify the technical and economic feasibility of a pilot scale production process for an integrated biorefinery using AFEX pretreatment. A preliminary design for an AFEX and CAFEX (continuous AFEX) has been completed (Appendix A). These activities are essential prerequisites to the construction and operation of a pilot plant to demonstrate the commercial potential of this technology. Findings from the FEED study were used to develop a proposal for the U.S. Department of Energy (DOE) supported by funding from the American Recovery and Construction Act of 2009 for the construction and demonstration of a fully integrated biorefinery pilot plant using AFEX pre-treated biomass. Funding would have moved the project from the *detailed investigation* stage to the *development phase* to further de-risk and scale up the technology. We learned in early December, 2009 that the \$23.5 million project was not funded.

From DOE reviewer comments and de-briefing and conversations with major industry players, a critical issue has emerged. Ammonia recovery is a key economic parameter that will drive the economic feasibility of an integrated biorefinery using AFEX pretreated biomass. Because ammonia recovery is a critical component of the process, it became clear that ammonia recovery would have to be demonstrated before moving on to the *development phase* and construction of a pilot scale plant. Potential industry partners such as ICM, The Andersons and Great River Energy have expressed interest in the technology. However under current economic conditions they are not willing to make the significant capital investment necessary to build a

pilot plant without demonstration of ammonia recovery. Because the overall business climate is more risk averse than 2-3 years ago at the onset of this project, without further de-risking by demonstrating ammonia recovery, investors are not willing to make the significant investment necessary to build a pilot scale plant.

To address this “chicken and egg” scenario, that is, a pilot plant is needed to demonstrate ammonia recovery, but commercial investors are not willing to build a pilot plant until ammonia recovery has been demonstrated, work has begun on an alternate means to demonstrate ammonia recovery. Based on preliminary projections, the cost for demonstrating ammonia recovery using this new approach will likely be far less than the \$23 million needed to build the fully integrated pilot plant. Work plans and cost estimates for this new approach are being developed and will be available in the coming months. Once work plans have been developed, efforts will begin to secure private, public or some combination of private and public funds for the ammonia recovery demonstration project.

As a result of the need to demonstrate ammonia recovery prior to the construction of a pilot plant, the *development phase* will likely take place in two stages. The first stage will involve demonstrating ammonia recovery using the new approach currently being developed. Once ammonia recovery has been demonstrated the commercialization process will resume and would involve identifying and securing a private sector partner(s) for the construction of a pilot scale plant. At the onset of this project it was thought that efforts to secure a commercial partner could begin at the end of the *detailed investigation*. However, with the additional requirement of demonstrating ammonia recovery, efforts to identify and begin conversations with potential investors would be premature. Accordingly a detailed strategic business plan aimed at identifying and securing a private sector partner(s) is impossible at this time.

## **LIKELY NATURE OF OPERATIONS**

Two options for commercialization can be envisioned 1) a centralized AFEX/biorefinery where the AFEX treatment facility is co-located at the bioconversion facility (saccharification and fermentation) and 2) a decentralized option where biomass processing and AFEX pretreatment are located separate from the bioconversion facility. Research has indicated that large biorefineries capable of handling 5,000 to 10,000 MT of biomass /day are preferable from an economic standpoint (Carolan, Joshi and Dale, 2007). However, large, biorefineries must contend with increased transportation costs and storage of low bulk density biomass.

The second option is a network of Regional Biomass Processing Centers (RBPC) that would form an extended supply chain for the bioconversion facility. Biomass would be collected and processed at these regional centers using the AFEX pretreatment process. The AFEX pretreatment process makes a stable intermediate material that can be stored with no deterioration of available sugars. Because lignin is moved to the particle surface in the AFEX process (Balan et al. 2006), the biomass is easy to densify without the use of steam or binders normally required for agricultural products (unpublished data, Dr. Bruce Dale, Michigan State University). AFEX treated biomass would be densified and stored on site providing a cost

advantage in terms of storage, handling and transportation. The ability to store and handle biomass using conventional delivery and receiving systems would help to alleviate some of the issues associated with storage, transportation and handling of low bulk density biomass.

An intermediate supply chain entity will likely be created to supply biomass to a centralized biorefinery. Because of the quantity of biomass needed to feed a commercial scale biorefinery (5,000 - 10,000 MT per day), the purchase, storage, transportation and delivery of biomass will be a significant undertaking. It is not likely the biorefinery operator will be inclined to nor have the capabilities to negotiate and contract with hundreds of individual producers and manage the logistics of biomass harvest, storage and transportation. An intermediate supply chain entity may not be as critical an element for regional biomass pretreatment centers. Depending on the scale of the pre-treatment centers, the pretreatment center may function as the intermediate supply chain entity contracting with multiple producers and supply biomass to the end user. Great River Energy, a Minnesota based electrical generation coop currently building a generation facility with capabilities of cofiring biomass, has explored issues related to biomass supply including the necessity of a supply chain entity (Great River Energy, 2009). Additional research is needed to address issues related to harvest, transportation, storage, densification, logistics, dedicated feedstock production, feedstock availability, contracts and producer relationships.

A centralized biorefinery will likely need to utilize various types of biomass. Because of the volume of biomass needed to fuel a commercial biorefinery, multiple feedstocks will likely be used. Harvest, transportation and storage of a single feedstock present numerous challenges. For example there is a very narrow window of opportunity to harvest corn stover. In recent years in North Dakota, weather conditions have delayed the corn harvest into November and December. In some cases harvest has been delayed until the next spring. Harvesting enough stover before the onset of winter to feed a commercial biorefinery in such a short period of time likely under less than ideal conditions may not be feasible. It is more likely that multiple feedstocks will be used. A combination of agriculture residue such as wheat straw and corn stover and dedicated energy crops with biomass harvest taking place at various times of the year may be required. Under a scenario where the biorefinery uses multiple feedstocks, AFEX pretreatment provides an advantage over other pre-treatments. AFEX is effective on multiple agricultural non-woody feedstocks and produces a stable intermediate product that can be stored, transported and integrated with subsequent processing.

Finally, first generation biorefineries will likely be co-located with an existing corn-based ethanol facility. This would reduce capital costs by integrating hydrolyzed and fermented biomass into the existing distillate system. Other advantages would include using existing systems for transportation and marketing the final product. Integrating first generation cellulosic ethanol production with an existing corn based facility would improve the overall economics of the facility.

## POTENTIAL MARKETS

Liquid fuels would be the primary product of the biorefinery. A freestanding commercial cellulosic ethanol plant using AFEX pretreatment is envisioned to produce 120 million gallons of ethanol per year. A significant component of the biorefinery would be its power source. Biomass consists of cellulose, hemicellulose and lignin. Lignin which cannot be converted into fermentable sugars will be burned to produce steam to power the biorefinery. Excess electrical power would be sold back to the grid, generating \$8.2 million in income per year. Not only does this approach improve the overall economics by using lignin to power to power the plant, electricity is a marketable output of the biorefinery.

Biomaterials are another potential product of a commercial biorefinery. Cellulose fibers have excellent mechanical properties and when combined with bio-based polymer may used to make biocomposites that could substitute for fiberglass and plastic in many applications including automotive, furniture, office/storage, marine, housing and recreation. Cellulose nanofibers for production of biocomposite materials can be a value-added byproduct in a cellulose to ethanol plant improving overall economic viability. Based on work completed by Dr. Larry Drzal, microfibrillated cellulose (MFC) appears to be more promising than cellulose nanowhiskers (CNW). MFC have similar properties but can be produced using a simpler process that costs less than producing CNF. Additional research is needed in this area. Good dispersion of the fibers is critical to producing a polymer with desirable mechanical properties. While preliminary studies appeared to have solved the dispersion issue, these results have not been replicated. Complete details of for findings related to cellulosic nanowhisiker technology can be found in Appendix B, Cellulose Nanowhisiker Technology Development.

Animal feed may also represent a potential market for Regional Biomass Processing Centers. Bench scale research has shown that AFEX treated materials has digestibility equal to corn grain for ruminant animals (Bals et al. 2009). Feeding trials are needed to assess the ability of various classes of ruminant to convert various types of AFEX treated biomass to energy.

AFEX pre-treated biomass may also potentially be used as fuel. Pelleted switchgrass is already being marketed as fuel for residential pellet stoves and cofired for commercial electricity generation in Missouri. Great River Energy has expressed an interest in potential for firing either AFEX pre-treated biomass or raw biomass in their co-fire generation facility currently under construction in Spiritwood, North Dakota. Characteristics of AFEX pre-treated material may make it a superior feedstock to untreated biomass. Additional research is required to determine if AFEX treated biomass would be preferred to untreated biomass for electricity generation. AFEX treated biomass can be used on multiple feedstocks and sold into several markets making it less vulnerable to the volatility of a single market. Multiple markets reduce risk and could enhance the prospects for rapid and broad commercial deployment.

## CAPITALIZATION REQUIREMENTS AND PROJECTED FINANCIAL PERFORMANCE

Capitalization requirements and financial performance are detailed in Appendix A. These estimates and the following discussion should be considered directional and assume ammonia recovery. The commercial biorefinery is projected to produce a return on investment greater than the cost of capital. A commercial plant selling ethanol for \$2.19 per gallon yields a 21.6 percent return on investment. The minimum ethanol price to meet a 9.02 percent return on investment would be \$1.58 per gallon. Total construction costs were estimated to be \$379 million. Life cycle analysis for an integrated biorefinery indicates a 65 percent reduction in greenhouse gas emissions and a 73 percent reduction in fossil fuel usage. Bench scale testing has demonstrated 94 percent of theoretical yield of fermentable sugars, 98 percent recovery of ammonia and 5 percent ethanol titer in high-solids enzymatic hydrolysis and fermentation of AFEX pre-treated corn stover. This data demonstrates that an AFEX integrated biorefinery has significant commercial potential.

### SUMMARY ACCOUNTING

#### Grant Details

NDSU Agribusiness & Applied Economics	
Personnel (salary, benefits and consulting services)	\$287,872
Travel, communications and supplies	
\$ 12,128	
MBI International	
Personnel (salary and benefits)	\$347,530
Material, supplies and equipment	\$ 70,000
Travel	\$ 12,470
Michigan State University	\$ 70,000
<b>Total</b>	<b>\$800,000</b>

#### Matching Funds

U.S. Department of Energy	\$250,000
USDA ARS	\$278,000
MBI International	\$150,000
Great River Energy (in-kind)	\$25,000
<b>Total</b>	<b>\$703,000</b>

#### Other Support

USDA CSREES	\$494,638
ND APUC	\$86,100
<b>Total</b>	<b>\$580,738</b>



## REFERENCES

Balan, V., Sh. Chundwat Sh., B. Bals, and B. Dale. 2006. The Case of Ammonia Fiber Expansion (AFEX). Presented at AIChE Annual Meeting. San Francisco, California.

Bals, B., H. Murnen, B. Dale, D. Main, and M. Allen. 2009. Food and Fuel: Integrating Animal Feed Production with Cellulosic Biofuels. Presented at the 31<sup>st</sup> Symposium on Biotechnology for Fuels and Chemicals. New Orleans, Louisiana.

Carolan, J., S. Joshi, and B. Dale. 2007. Technical and Financial Feasibility Analysis of Distributed Bioprocessing Using Regional Biomass Pre Processing Centers. Journal of Agricultural and Food Industrial Organization, Volume 5, Article 10 (2007) SPECIAL ISSUE: Explorations in Biofuels Economics, Policy, and History.

Great River Energy. 2009. Study of the Feasibility of a Biomass Supply for the Spiritwood Industrial Park. A Report to the North Dakota Industrial Commission.  
<http://www.nd.gov/ndic/renew/projects/r-001-003sp.pdf> (viewed January 22, 2010).

## **Appendix A**

### **“Front End Engineering and Design of an AFEX Integrated Biorefinery”**

## FRONT END ENGINEERING AND DESIGN OF AN AFEX INTEGRATED BIOREFINERY

**Subaward No. FAR0013936**

**under NDSU Prime BM001-001**

MBI Project No. 1622.001

### **FINAL REPORT**

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## Objectives

The goal of this project was to complete a front end engineering and design (FEED) study for a pilot scale plant to demonstrate the commercial potential of this technology, a critical step in establishing a biomaterials industry in North Dakota. Initial efforts were focused on technical and economic requirements for commercializing technology to produce bio-based cellulose nanowhiskers. Specific objectives include:

1. Completing the detail investigation necessary to define:
  - Scalable process design
  - Mass and energy balances necessary to determine the cost of the process
  - A procedure for qualitative and quantitative analysis of the structural materials available from wheat straw
  - A system for analyzing the structural enhancements of polymers from the inclusion of wheat straw fibers
2. Refining the initial investment analysis for the business as data is added to key parameters regarding capitol costs and manufacturing yields.
3. Preparing of a strategic business plan for integration of public and private sector resources to provide investment for pilot plant construction and, when appropriate, construction of commercial manufacturing facilities. The strategic business plan will detail the likely nature of operations of a corporate entity as well as examine potential markets, capitalization requirements, and project financial performance.

## Summary

MBI's original concept for continuous Ammonia Fiber Expansion (AFEX) treatment (CAFEX-I) of biomass was based on wood delignification processes in the pulp and paper industry, using a Pandia-type reactor with feed and discharge screws. The advantage of the CAFEX-I approach is that it relies on equipment which has been in use for decades, and has been shown to be effective for a variety of biomass feedstock materials. The operating principles of plug screws and Pandia reactors are largely independent of the fluid properties of the feedstock materials, so that the same equipment can be used to process materials with different properties, with only minor modifications. The primary disadvantage of the CAFEX-I approach is the high capital cost of the equipment required. It is unclear whether a CAFEX-I reactor system could be cost competitive with other leading biomass pretreatments.

Due to concerns over the high equipment costs associated with the CAFEX-I approach, MBI investigated methods for accomplishing the same process operations with lower-cost equipment. The result was the CAFEX-II concept, which uses a progressive cavity pump to charge moist biomass into a pressurized device for mixing with ammonia and steam. The use of a progressive cavity pump is central to the CAFEX-II concept. However since wheat straw could not be pumped with any progressive cavity pump at any moisture level or particle size, we are unable to process wheat straw in the CAFEX-II system.

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In May of 2009, the U.S. Department of Energy released a Funding Opportunity Announcement, DE-FOA-0000096, titled "Recovery Act – Demonstration of Integrated Biorefinery Operations." At that time, the CAFEX-II system was incapable of processing high-impact feedstocks such as wheat straw and corn stover, due to the progressive cavity pumping problems. It was therefore decided to apply for funding to build and operate a CAFEX-I pilot plant, under Topic Area 2 of the FOA. The application includes detailed design information, mass and energy balances for both pilot- and commercial-scale operation of CAFEX plants, as well as techno-economic analysis of a commercial ethanol plant using AFEX treatment.

The compositional, morphological, and surface chemical changes introduced into wheat straw as a result of AFEX and Simultaneous Saccharification and Fermentation (SSF) treatments were studied. Cellulose nanowhiskers (CNW) and Microfibrillated cellulose (MFC) were extracted from this ethanol production residue at reasonable yields. In order to produce a totally biobased composite material, several methods to combine the wet wheat straw based MFC and CNW with Poly Lactic Acid (PLA) were investigated at concentrations up to about 5 wt% including an new emulsification-freeze drying process, filtration and lamination and spray processing.

Mechanical properties of the composites did not show improvement consistent with the values expected for a reinforcement with properties similar to MFC and CNW. The lack of measurable increases in the composite properties has been attributed to poor dispersion within the PLA matrix.

Good dispersion of the cellulose nanofibers is a key to producing a polymer composite with high mechanical properties and to fully realize their potential as a reinforcing material. Although some preliminary studies had appeared to show that the Emulsion-Filtration technique developed had solved the fiber aggregation problem, after switching to a new source of PLA resin, we have not been able to achieve the same degree of dispersion so far. Further research is necessary in order to identify a processing method that provides good dispersion of the MFC in PLA or other water based polymers.

The fermentability of sugars derived from AFEX-treated wheat straw was assessed using *Zymomonas mobilis* 8b. Ethanol yield based on the available glucose and xylose in the wheat straw was about 62%. There was no sugar residue at the end of the fermentation, which indicates that *Z. mobilis* 8b was able to metabolize both glucose and xylose derived from AFEX-treated wheat straw.

Different combinations of cellulase and xylanase were used in hydrolysis of AFEX-treated wheat straw to determine the most effective enzyme mixture. This experiment showed that it is possible to lower the total amount of enzyme without compromising hydrolysis yield and confirmed that having the right enzyme combination is very critical to reduce the cost of biomass conversion.

## Approach

Our approach assumes that cellulose nanofibers for production of biocomposite materials can be a value-added byproduct in a cellulose-to-ethanol biorefinery. Production of cellulosic ethanol from

wheat straw is dependent on the effects of the Ammonia Fiber Expansion (AFEX) pretreatment process for conversion of cellulosic biomass to fermentable sugars. The AFEX process has been licensed to MBI for development and commercialization. The residual materials from ethanol fermentations contain cellulose nanofibers. The technical approach in this project period addressed the following areas:

**1. Detailed investigation of the AFEX process with regard to processing of wheat straw for conversion of cellulose/hemicellulose to ethanol. The program will seek to optimize the AFEX process for efficient conversion of wheat straw cellulose and hemicellulose to fermentable sugars for production of ethanol and define the mass balances.**

**Key processes to be investigated are:**

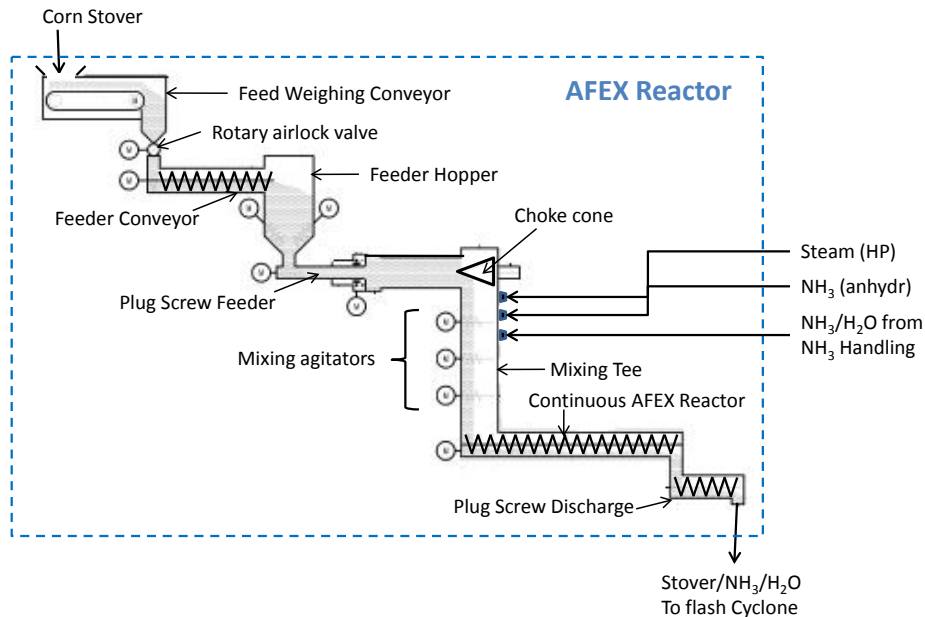
- **Verify the scalability of a continuous AFEX process design**

Continuous AFEX (CAFEX) approaches – CAFEX-I and -II

Any process for continuous AFEX (CAFEX) treatment of wheat straw will necessarily include operations such as continuous charge and discharge of feedstock into and out of a pressurized reactor vessel, and a reactor vessel that provides adequate residence time for contacting the biomass with ammonia. Economic operation of a CAFEX process will also require operations for recovery and re-use of ammonia. Investigations of various CAFEX process designs have led to two distinct process concepts, which for convenience are referred to as CAFEX-I and CAFEX-II.

CAFEX-I

MBI's original concept for continuous AFEX treatment of biomass was based on wood delignification processes in the pulp and paper industry. In pulp processing, wood chips are fed into pressurized delignification reactor vessels using plug screw feeders. Plug screw feeders are manufactured by various vendors, with some variation in design between vendors, but generally use a motor-driven tapered screw to continuously force feedstock material against a tapered orifice or cone, which compresses the material into a plug. As the plug is forced through the orifice it breaks apart into an expanding chamber, which may be held under pressure by the seal formed by the material plug. Once charged into the pressurized reactor, the material can then be discharged by means of a similar plug screw. The residence time of the material within the pressurized reactor can be controlled by various mechanisms. Pandia-type reactors, which use a horizontal or slightly inclined auger to transport material through a pipe, are widely used in pilot-scale pulp processing operations. Figure 1 shows a diagram of a Pandia reactor with feed and discharge plug screws. CAFEX-I refers to the use of a Pandia-type reactor with feed and discharge screws adapted for continuous AFEX treatment of biomass.



**Figure 1.** Continuous AFEX reactor system.

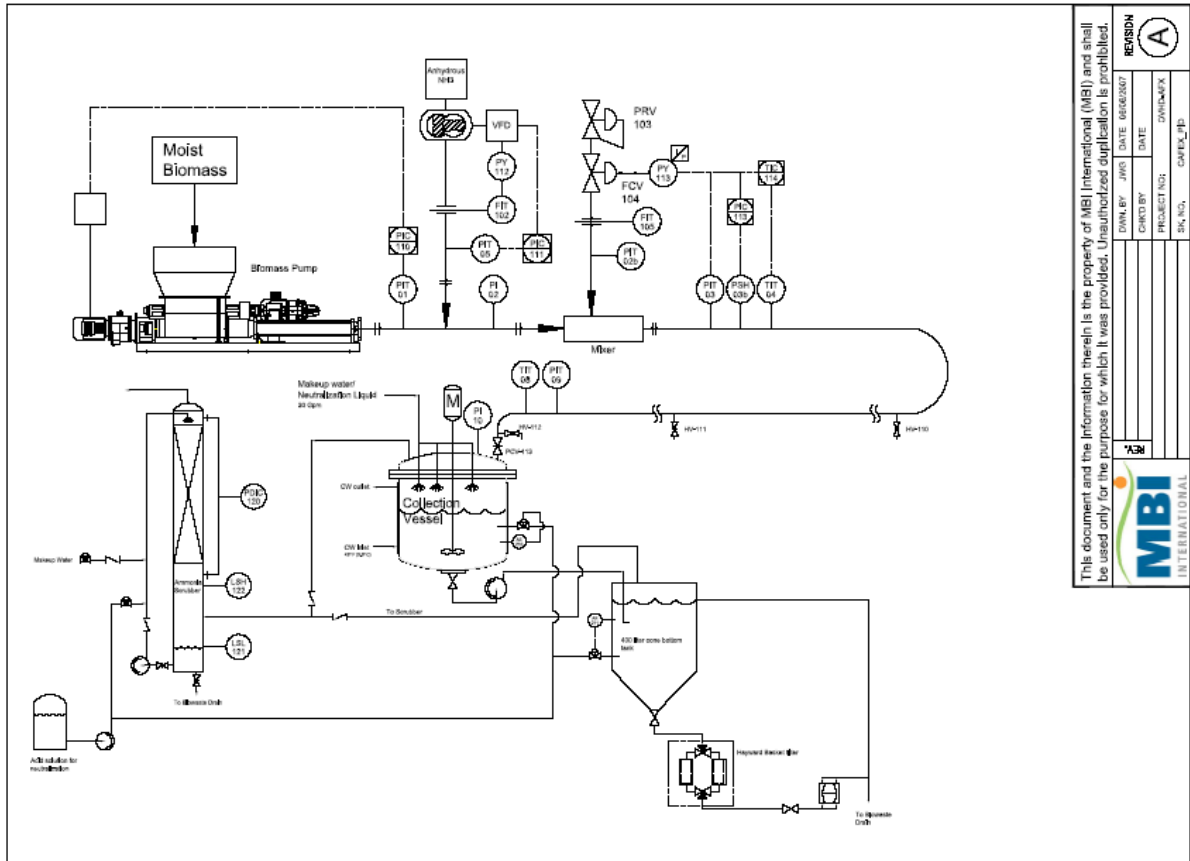
The advantage of the CAFEX-I approach is that it relies on equipment which has been in use for decades, and has been shown to be effective for a variety of biomass feedstock materials. The operating principles of plug screws and Pandia reactors are largely independent of the fluid properties of the feedstock materials, so that the same equipment can be used to process materials with different properties, with only minor modifications. Appendix A, attached to this report, includes a letter from Andritz Mechanical Pulping Division, a leading manufacturer of equipment for the pulp and paper industry, stating that their plug screw pressure feeder, horizontal reactor, and discharge device have been shown to meet AFEX processing conditions using various feedstock materials, including wheat straw and corn stover. The primary disadvantage of the CAFEX-I approach is the high capital cost of the equipment required. It is unclear whether a CAFEX-I reactor system could be cost competitive with other leading biomass pretreatments.

#### CAFEX-II

Due to concerns over the high equipment costs associated with the CAFEX-I approach, MBI investigated methods for accomplishing the same process operations with lower-cost equipment. The result was the CAFEX-II concept, which uses a progressive cavity pump to charge moist biomass into a pressurized device for mixing with ammonia and steam. Residence time of the biomass/ammonia/ steam mixture under AFEX temperature and pressure conditions is then provided using a simple tubular reactor. Discharge from the reactor tube can be controlled using a valve or a positive displacement pump. A CAFEX-II reactor system was designed and constructed in MBI's pilot plant. Figure 2 shows a schematic

of the CAFEX-II system. Shakedown of this system, including demonstration of adequate temperature and pressure control, has been completed using DDGS feedstock.

The advantage of the CAFEX-II approach is that it uses equipment that is significantly less costly than CAFEX-I. The primary disadvantage of the CAFEX-II approach is that the progressive cavity pump has not been tested for feeding a variety of feedstock materials under pressure.



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**Figure 2. Schematic of CAFEX II Pretreatment System**

### Wheat straw pumping using progressive cavity pumps

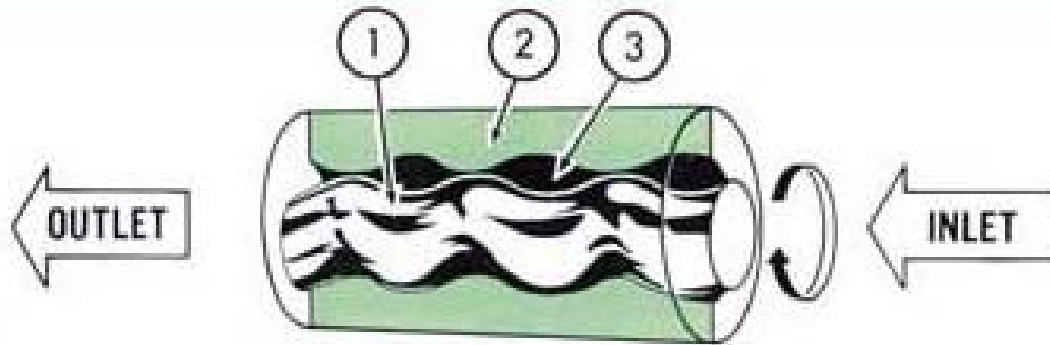
The use of a progressive cavity pump is central to the CAFEX-II concept. Progressive cavity pumps are manufactured by many different vendors, and have been used for decades to pump liquids containing suspended solids, as well as slurries with high solids content. In progressive cavity pumping, as shown in Figure 3, rotation of a helical steel rotor is driven by an electric motor. The rotor turns within an elastomeric stator tube which has a helical internal channel. A series of discrete cavities are formed in the annular gap between the rotor and stator; rotor rotation causes these cavities to progress axially from the inlet to outlet of the pump element, thereby conveying feed material. Volumetric capacity

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depends on the cavity volume and rotor speed, while the maximum differential pressure developed by the pump depends on the number of helical stages within the element.



**Figure 3 – Diagram of a progressive cavity pump element showing rotor (1), stator (2), and cavities (3). (From [www.process-controls.com](http://www.process-controls.com))**

Progressive cavity pumping action requires flow of feedstock material into the cavity as it forms at the inlet end of the pump element. This requirement limits the application of progressive cavity pumping to materials with adequate fluid properties. Many commercial progressive cavity pump models also include an auger that continually conveys feed material from a hopper toward the pump inlet. The feed auger helps to force-feed material into the inlet cavity of the pump element, but materials that resist intrusion into the inlet cavity will not be pumped, despite the auger action.

The fluid properties of moist wheat straw depend primarily on the moisture content and particle size distribution. Table 1 gives water absorption capacities and hydraulic conductivities of wheat straw and other biomass ground to 1 and 2 mm average particle size. It should be noted that AFEX processing of biomass with moisture greater than 80 mass% would be uneconomical due to the high ammonia loading required. However, wheat straw with moisture less than 80 mass% is not saturated, and therefore does not behave as a fluid, but rather as a bed of particles. The hydraulic conductivity of a bed of particles is the proportionality constant in Darcy's Law, and is a measure of the ease with which fluid flows through the bed. The hydraulic conductivities of wheat straw particle beds are quite high, around  $0.038 \pm 0.001$  cm/s, and this high conductivity turns out to have serious implications for progressive cavity pumping.

**Table 1 – Properties of biomass**

Biomass	Particle Size  (mm)	Water Absorption  Saturated Capacity  (mass%)	Hydraulic conductivity (k)  (cm/s)
Wheat straw	1	87	0.039
Wheat straw	2	86	0.037
Corn stover	1	86	0.0022
Corn stover	2	84	0.0023
Corn fiber	1	65	0.0012
Corn fiber	2	65	0.0074
DDGS	1	61	0.0138

As described above, most progressive cavity pump designs use a feed auger to force feedstock material toward the cavity at the inlet of the pump element. When moist wheat straw is forced by the auger toward the pump element inlet, it becomes compressed into a bed of particles, which releases free water. The high hydraulic conductivity of the particle bed means that the inlet cavity of the pump element can easily draw the free water in, leaving the solid particles behind. This “de-watering” behavior is a common failure mode for progressive cavity pumping of fibrous biomass. Wheat straw pumping tests using various models of Seepex progressive cavity pumps, including BTI 5-24, BTI 17-12, and BTH 17-12 models, all showed de-watering with little or no solids throughput, regardless of moisture content or particle size. For comparison, DDGS do not de-water, due in part to their lower hydraulic conductivity.

MBI has contacted progressive cavity pump manufacturers Seepex Inc. and Moyno Inc. to discuss pumping of high-impact biomass such as wheat straw. The pump manufacturers are aware of the de-watering problem, but to date have no solutions. MBI has also investigated the use of additives, including surfactants, sodium hydroxide and other bases, and heating to modify the properties of wheat straw feeds. While some of these modifications did reduce the extent of de-watering, none provided adequate pumping performance for CAFEX-II processing. Because wheat straw could not be pumped with any progressive cavity pump at any moisture level or particle size, we are unable to process wheat straw in the CAFEX-II system.

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In May of 2009, the U.S. Department of Energy released a Funding Opportunity Announcement, DE-FOA-0000096, titled “Recovery Act – Demonstration of Integrated Biorefinery Operations.” At that time, the CAFEX-II system was incapable of processing high-impact feedstocks such as wheat straw and corn stover, due to the progressive cavity pumping problems. It was therefore decided to apply for funding to build and operate a CAFEX-I pilot plant, under Topic Area 2 of the FOA. MBI’s application is attached to this report as Appendix B. MBI’s application was based on corn stover processing. However, the small differences in the properties of corn stover and wheat straw would have little effect on the operation of a CAFEX-I plant. The application includes detailed design information, mass and energy balances for both pilot- and commercial-scale operation of CAFEX plants, as well as techno-economic analysis of a commercial ethanol plant using AFEX treatment.

**• AFEX processing conditions including temperature, pressure, ammonia loading and retention times**

The most effective AFEX conditions for pretreatment of wheat straw were previously identified using our 1 gallon AFEX reactor. The conditions were chosen based on the highest sugar yields obtained in enzyme hydrolysis of AFEX-treated wheat straw. As part of the presenting work these conditions (temperature:90°C, ammonia loading: 1 kg ammonia per kg of dry biomass, 60% moisture content and 30 min residence time) were verified in our 5 gallon AFEX reactor. Several AFEX runs with wheat straw were carried out in the 5 gallon reactor under the conditions stated above. The performance of the process was evaluated via enzyme hydrolysis. As it is clear from data presented in Table 2 both reactors demonstrate similar performance. The generated AFEX-treated wheat straw were collected and stored in cold room for further use in hydrolysis and fermentation experiments.

**Table 2.** Hydrolysis of AFEX-treated wheat straw.

Biomass	ID	Reactor	Temperature, °C	Ammonia: biomass	Time, min	Moisture content %	Glucose yield%	Xylose yield%
Untreated wheat straw							24±2	6±2
AFEX treaded wheat straw	853-3A, 3B	1 gallon	90	1:1	30	60	60±2	52±2
AFEX treaded wheat straw	961-12A-14B	5 gallon	90	1:1	30	60	61±3	53±2

Hydrolysis were carried out for 72 hr with 15 FPU of cellulase per gram of cellulose. Yields were calculated based on the available sugars in the biomass. Composition is listed in Table 3.

**Table 3.** Carbohydrate composition of untreated wheat straw based on dry weight

Biomass	% Glucan	% Xylan	% Galactan	% Arabinan	% Mannan
Untreated wheat straw(average of two different batches)	32.43±2	18.46±0	2.24±0.3	2.87±0.5	2.05±1

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- **Enzyme hydrolysis conditions including screening of enzymes for more efficient conversion**

Maximal utilization of all the biomass polymeric sugars is essential to make the economics of biomass processing feasible. Existing cellulase mixtures have been developed to hydrolyze mostly acid/high temperature treated biomass and are not optimal for AFEX-treated material. AFEX-treated biomass, unlike acid treated material, have significant amount of hemicellulose that needs to be hydrolyzed to monomeric sugars. Therefore complete and balanced cellulolytic and xylanolytic systems are required to achieve maximum hydrolysis of plant cell wall polymers at minimal cost. It has been shown that the hydrolysis of glucan and xylan are intimately linked and whatever enhances glucan conversion also tends to increase xylan conversion (and vice versa). It is important to provide not only adequate total hemicellulase activity but also the correct distribution of enzyme activities between cellulase and hemicellulase mixture. In this work AFEX-treated wheat straw was hydrolyzed with different combinations of cellulase and xylanase to maximize both glucose and xylose yield. Recently Novozyme has launched two new enzymes, one cellulase called Ctec and one hemicellulase called Htec. During this reporting period we received samples of these two enzymes and evaluated their performance in hydrolysis of AFEX-treated wheat straw. Table 4 summarizes hydrolysis results of AFEX-treated wheat straw hydrolyzed with different combination of enzymes. Hydrolyses were performed following National Renewable Energy Laboratory (NREL) lap-009 protocol.

The cost of the cellulase enzyme is one of the major contributors to the overall ethanol production cost. It is obvious that any reductions in this cost have a significant effect on the total operating cost. Using the right combinations of enzyme will allow us to reduce the overall enzyme usage. Some of the experiments shown in Table 4 were carried out to identify the lowest enzyme (total amount of protein) loading that can be used in hydrolysis of AFEX-treated wheat straw without significantly compromising the conversion yield.

**Table 4.** 72 hr hydrolysis of AFEX-treated wheat straw with different enzymes. Hydrolysis was performed in duplicate and results are presented as the mean value (experiment ID# 948-92)

	FPU of ctec/ g glucan	mg of Ctec Protein	mg of Htec Protein	mg of Novo 188 protein	Total mg of protein	Glucose yield %	Xylose yield%
1	5	4.56	2.736	0	7.296	41.22 ±0.5	49.64 ±2
2	5	4.56	3.42	0	7.98	38.29 ±0.5	46.32 ± 0.5
3	10	9.12	0	0	9.12	24.08 ±1	26.77 ± 2
4	10	9.12	1.368	0	10.488	50.81 ±2	49.71 ±2
5	10	9.12	2.736	0	11.856	51.49 ±1	50.51 ±0.5
6	15	13.68	0	0	13.68	29.48 ±1	30.95 ±1
7	15	13.68	1.368	0	15.048	58.23 ±0.3	53.53 ±1
8	15	13.68	2.736	0	16.416	58.05 ±0	52.81±0.5
9	7.5	7.6	6.08	0	13.68	48.64 ±1	48.98 ±2
	FPU of Spezyme Cp/ g glucan	mg of Spezyme Cp protein	mg of Htec Protein	mg of Novo 188 protein	Total mg of protein	Glucose yield %	Xylose yield%
10	15	13.9	2.73	10 (~42 CBU/g cellulose)	26.63	72.5 ±0.5	63.99 ±0.5
11	10	9.8	2.73	10 (~42 CBU/g cellulose)	22.53	65.57 ±1	61.39 ± 0.5
12	15	13.9	0	10 (~42 CBU/g cellulose)	23.9	61.2 ±1	53.3 ±2
13	10	9.8	0	10 (~42 CBU/g cellulose)	19.8	52.03 ±1	52.16 ±0.5

Two different cellulases, Spezyme Cp from Genencor and Cellic Ctec from Novozyme, were used in this experiment. Activity of the enzymes was measure following NREL Lap-006 procedure. Protein content of the enzyme products was provided by the manufacturers. Information provided by Novozyme indicates that Ctec enzyme contains enough  $\beta$ - glucosidase activity; therefore hydrolyses with Cellic Ctec were not supplemented with Novo 188. For further confirmation experiments were set up for hydrolysis of Avicel (pure cellulose) using Ctec with and without Novo 188. As it is clear from the data presented in Table 5, adding Novo 188 not only did not help it actually reduced the glucose yield. On the other hand since Spezyme Cp does not contain enough  $\beta$ - glucosidase activity, Novo 188 was added to all of the hydrolysis performed with Spezyme Cp.

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**Table 5.** 72hr hydrolysis of Avicel using Ctec with and without Novo 188. Hydrolysis was performed in duplicate and results are presented as the mean value (experiment ID# 949-83)

Ctec loading	Novo 188	Glucose yield%
15 FPU/ g cellulose	42 CBU/g cellulose	72% ±0
15 FPU/ g cellulose	0	74% ±0

Comparing the performance of Spezyme Cp and Ctec enzyme at the same level of loading (FPU/g cellulose) suggests that Spezyme Cp combined with Novo 188 provides a more effective enzyme combination for hydrolysis of AFEX-treated wheat straw.

Apparent from data presented in Table 4, it is possible to lower the total amount of enzyme in hydrolysis of AFEX-treated wheat straw without compromising hydrolysis yield (comparing experiment # 11 and 12, experiment #4 and 6). These data confirmed that having the right enzyme combination is very critical to reduce the cost of biomass conversion (comparing experiment #6 and 9). These data show that cellulase and xylanase activities can be optimized to reduce the total enzyme loading in enzymatic hydrolysis of AFEX-treated biomass.

- **Screening of organisms capable of utilizing both 5 and 6 carbon sugars**

When choosing a microorganism for fermentation, several important attributes should be considered, including yield, ethanol tolerance, productivity, and growth requirements. Among these traits, ethanol yield has the most impact. If ethanol yield is high, less feedstock would be needed to produce the same amount of ethanol. Based on this requirement, *Zymomonas mobilis*, which has demonstrated highest ethanol yield on sugar complex containing glucose, ( Lee, K. J., et al., *Biotechnology letters*, 1980. 2(11): p. 487-492; Rogers, P. L., et al., *Process Biochemistry*, 1980, 15(6): p. 7-11; and Rogers, P. L., et al., *Adv. Biotechnol.*, [Proc. Int. Ferment. Symp.] 6th, 1980,) has become one of the most promising microorganisms for ethanol production.

Even though *Z. mobilis* has unique metabolism and ability to rapidly and efficiently produce ethanol from simple sugars, it has not been used commercially for several reasons. One of the major issues is that *Z. mobilis* typically only uses glucose, fructose and sucrose as their substrates. Since pentoses such as xylose is a major component of hemicellulose in most biomass feedstock such as wheat straw, it is usually essential for a fermenting microorganism to use this sugar in ethanol production for a good product yield from biomass. Fortunately metabolic engineering has been successfully applied to develop a *Zymomonas* strain to ferment xylose (Zhang, M., Engineering *Zymomonas mobilis* for efficient ethanol production from lignocellulosic feedstocks. ACS national meeting, 2003 and U.S. Pat. No. 7,223,575), and as well as arabinose (Mohagheghi, A., et al., *Applied biochemistry and biotechnology*, 2002, 98-100: p. 885-898). By genetic engineering technology, engineered *Z. mobilis* could potentially use all sugars present in most biomass feedstock.

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In this study the fermentability of sugars derived from AFEX-treated wheat straw was assessed using *Z. mobilis 8b*. This strain has been developed by NREL and it is capable of using glucose, xylose and arabinose for production of ethanol. The detail of the fermentation is described below.

Two fermentations (experiment ID# 948-90 and 91) at 15% solid loading using AFEX-treated wheat straw and *Z. mobilis 8b* were performed. The fermentations were carried out as Separate Hydrolysis and Co-Fermentation (SHCF) in 7L BiofloIII fermentors. The hydrolysis portion was carried out at 50°C and at pH 4.8 for approximately 72hr. An enzyme cocktail was made for each fermentor in 200 ml of water. Spezyme CP was added in an amount corresponding to 20 FPU/g cellulose, Novo 188 was added in an amount corresponding to 42 CBU/g glucan and plus 10ml of Multifact xylanase (from Genencor). The enzyme cocktail was filter sterilized with a 50mm pre-sterilized 0.2µm filtration unit. The fermentation process began when the fermentor temperature was lowered to 32°C, pH was adjusted to 6.0 using KOH, and the fermentors were inoculated with *Z. mobilis 8b*. The initial OD in each fermentor was about 1.5. Seed and fermentation preparation were carried out according to methods suggested by Mohagheghi et al (Mohagheghi, A., et al., Biotechnology Letters, 2004, 26: p. 321-325)

In an effort to minimize the possibility of contamination, it was decided to take only one sample during the hydrolysis period (at 72hr time point). During the fermentation period samples were taken every 24 hr. Samples were analyzed for sugars and ethanol content using HPLC system. Both fermentations were successful and no sign of contamination was observed. The results showed that sugars generated from AFEX-treated wheat straw were fermentable and HPLC results indicated that nearly all of the glucose and xylose were consumed in the first 24 hr of fermentation. Ethanol yield based on the available glucose and xylose in the wheat straw was about 62%. There was no sugar residue at the end of the fermentation, which indicates that *Z. mobilis 8b* was able to metabolize both glucose and xylose derived from AFEX-treated wheat straw. The Table 6 summarizes the hydrolysis and fermentation results obtained from this experiment.

<b>Table 6.</b> Hydrolysis and fermentation results for the fermentation with 15% solid loading AFEX-treated wheat straw.				
	<b>Glucose* Yield%</b>	<b>Xylose* Yield%</b>	<b>Ethanol g/l</b>	<b>Ethanol* yield%</b>
<b>After 72hr of hydrolysis</b>	65.95±1	50.5±2	0	0
<b>After 48hr of fermentation</b>			25.2±1	61.6±2

Yields are calculated based on the available glucose and xylose. Presented results are the mean value of the duplicate runs (two fermentations).

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## 2. Determine the best extraction methods for refining cellulose nanofibers from wheat straw fermentation residues

For production of AFEX-treated wheat straw fermentation residue, two fermentations (experiment ID#881-50(1&2)) were carried out. These fermentations were executed according to NREL Lap 008 protocol with some modifications. The detail procedure is provided below.

### Fermentation procedure:

Enzyme preparations:

An enzyme cocktail was made for each fermentor in 150 ml of water. Spezyme CP was added in an amount corresponding to 15 FPU/g cellulose, Novo 188 was added in an amount corresponding to 42 CBU/g glucan. The enzyme cocktail was filter sterilized with a 50 mm pre-sterilized 0.2  $\mu\text{m}$  filtration unit ([www.nalgenelabware.com](http://www.nalgenelabware.com)).

Medium and inoculum preparations:

*Saccharomyces cerevisiae* D5A was originally obtained from NREL, and stored frozen in glycerol at  $-70^{\circ}\text{C}$ . The medium used in the SSF fermentations contained corn steep liquor (CSL) 5g/L,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  0.62 g/l,  $\text{NH}_4\text{SO}_4$  2 g/l. The inoculum was grown in the same medium as used in the SSF fermentations, but with 2% w/v glucose as substrate. The inoculum was grown in 500 ml baffled Erlenmeyer flasks containing 150 ml of medium each. The flasks were incubated at  $30^{\circ}\text{C}$  and 150 rpm in a Model G25 incubator shaker (NBS, Edison, NJ). The inoculum was prepared in two stages. In the first stage a 150 ml culture was inoculated from a glycerol vial and grown over night. In the second stage 150 ml cultures were inoculated for each fermentor with 0.5ml of the overnight culture. The growth in the inoculum flasks were monitored by measuring the optical density. They were used to inoculate the fermentors when the  $\text{OD}_{600}$  reached 2.5-3.5, after about 14-15 hours of incubation.

Bioreactor preparations:

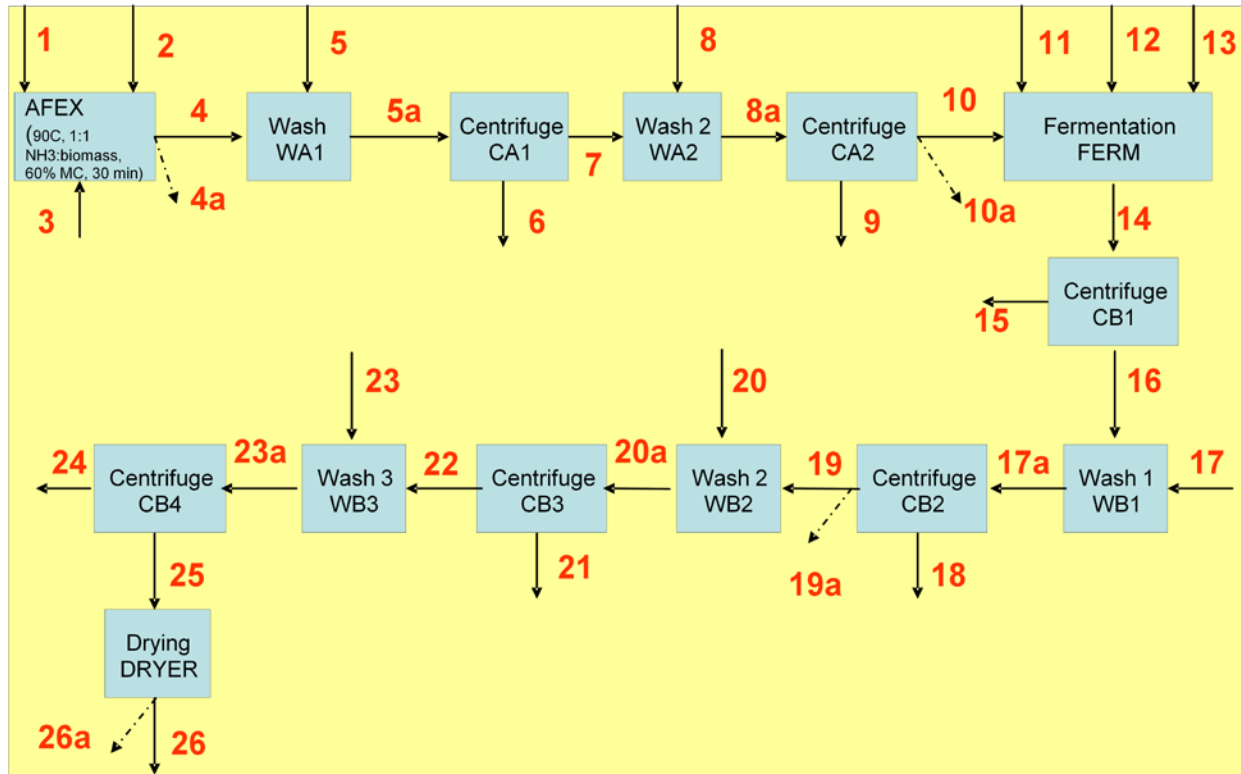
The amount of AFEX-treated wheat straw necessary to give 10% solids loading in a 5 liter volume was placed into the 7L BioFloIII fermentor (NBS, Edison, NJ), with the required amount of water containing the CSL and the medium salts. The fermentations were carried out as simultaneous saccharification and fermentation (SSF) in duplicates. As it was instructed by the MSU team (Dr. Drzal's group), who were extracting micro cellulose fiber from the fermentation residue, the AFEX-treated wheat straw was washed prior to the fermentation step. The fermentors were autoclaved for 1 hour. After autoclaving, the fermentors were operated at  $30^{\circ}\text{C}$ , 500 rpm, and pH 5.0. The pH was controlled through the automatic addition of 1M NaOH, and 4N  $\text{H}_2\text{SO}_4$ . The pH of the fermentation was monitored by checking the sample pH using an externally calibrated electrode, and adjusting the BioFloIII automatic pH controller as necessary. The SSF fermentation was started by adding 200 ml of enzyme cocktail and 150



ml of *S. cerevisiae* D5A inoculum. The initial OD<sub>600</sub> in each fermentor was about 0.5. Fermentation was carried out for 72 hr and every 24 hr samples were taken for sugar and ethanol analysis. At the end of the fermentation whole broth was collected and weight and volume were measured. Fermentations were carried out in duplicate. Both fermentations were successful and there was no sign of contamination. At the end of the fermentation there was no glucose left, which indicates the sugar generated from AFEX-treated wheat straw were fermentable. Ethanol yield based on the available glucose was about 45%.

Figure 4 is a flow diagram that shows the processes and the steps that have been followed to prepare AFEX-treated wheat straw fermentation residue for extraction of nano cellulosic fiber. The descriptions of the streams shown in Figure 4 are summarized in Table 7. A mass balance was constructed around the processes involved in the production of the fermentation residue. The details of the mass balance are summarized in Table 8. The mass balance was performed by measuring the composition, moisture content and the amount of each stream shown in Figure 4. The compositions and moisture content were measured according to NREL procedures (Laps 002 and 010 respectively). As it is clear from the data presented in Table 8 mass balance closure was achieved for each process.

**Figure 4 .** Flow diagram for production of AFEX-treated wheat straw fermentation residue



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**Table 7.** Description of the streams shown in Figure 4

<b>Stream #</b>	<b>Description</b>
1	2500 gram water
2	3750 gram ammonia
3	3750 gram dry wheat straw
4	1500 gram dry AFEX-treated wheat straw
5	15000 gram water, at room temperature, for 30 min.(this was done in 2 batches)
6	For fermentor #1: 5820ml collected wash 1, 0.845% solids For fermentor #2: 5700 ml collected wash 1, 0.845% solids
7	For fermentor #1: 2.2K gram collected solid 1, 69.8% moisture For fermentor # 2: 2.2KG collected solid 1, 65.8% moisture
8	15000 gram water, at tap DI °C, for how long 30 min.(this was done in 2 batches)
9	For fermentor #1: 6680 ml collected wash 2, 0.216% soild For fermentor #2 : 7320 ml collected wash 2, 0.216% solid
10	For fermentor #1: 2616 gram collected solid 2, 70.3% moisture For fermentor#2: 2120 gram collected solid 2, 71.9% moisture
11	2669.9 ml media, total liquid in fermentaion 5000ml
12	500 ml Enzyme 57.08 g Spezyme CP, 27.86 g Novo
13	300 ml Yeast inoculum
14	5181.46 gram of whole broth
15	3920 ml liquid broth collected 2.74% solids
16	1241.76 gram fermentation residue collected, 83.5% moisture content
17	731 gram water, at 50°C, for 30 min
18	839.16 gram collected post fermentation wash 1, 1.70% solid
19	680.95 gram collected post fermentation solid 1, 81.85% moisture (#3-#6)
20	245.02 gram water, at 50°C, #1 & #2 added together
21	gram collected post fermentation wash 2, 0.846% solid
22	374.81 gram collected post fermentation solid 2,
23	331.5 gram water, at 50°C
24	774.3 gram collected post fermentation wash 3, 0.382% average solid
25	289.63 gram collected post fermentation solid 3,
26	53.54 gram collected final sample,

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**Table 8.** Mass balance for the processes shown in Figure 4

Block	stream ID	amount (g)	Feed stream	Process stream	Removed stream	total in (g)	total out (g)	lost/gain	lost/gain (wt%)
AFEX	1	2,500	water			10,000	10,000	-	
	2	3,750	NH3						
	3	3,750	dry wheat straw						
	4	1,500		treated wheat straw					
	4a	8,500			treated wheat straw				
WA1	4	1,500		from AFEX to WA1		16,500	16,500		
	5	15,000	water						
	5a	16,500		from WA1 to CA1					
CA1	5a	16,500		from WA1 to CA1		16,500	15,941	(559)	-3.4%
	6	11,541			liquid after CA1				
	7	4,400		from CA1 to WA2					
WA2	7	4,400		from CA1 to WA2		19,400	18,571	(829)	-4.3%
	8	15,000	water						
	8a	18,571		from WA2 to CA2					
CA2	8a	18,571		from WA2 to CA2		18,571	18,571	-	
	9	13,835			liquid after CA2				
	10	1,730		from CA2 to Ferm					
	10a	3,006			from CA2 to Ferm				
FERM	10	1,730		from CA2 to Ferm		5,500	5,181	(319)	-5.8%
	11	2,970	media						
	12	500	enzyme						
	13	300	yeast						
	14	5,161		from Ferm to CB1					
CB1	14	5,161		from Ferm to CB1		5,161	5,189	28	0.5%
	15	3,948			liquid after CB1				
	16	1,242		from CB1 to WB1					
WB1	16	1,242				1,973	1,973	-	
	17	731	water						
	17a	1,973							
CB2	17a	1,973		from WB1 to CB2		1,973	1,876	(97)	-4.9%
	18	839.16			liquid after CB1				
	19	110.84		from CB2 to WB2					
	19a	925.90			WB1/CB2 to dryer and storage				
WB2	19	110.84				1,449	1,449	-	0.0%
	20	1338.35	water						
	20a	1,449.19		from WB2 to CB3					
CB3	20a	1,449.19				1,449	1,449	-	0.0%
	21	959.17			liquid after WB2/CB3				
	22	490.02							
WB3	22	490.02		from CB3 to WB3		1,170	1,170	-	0.0%
	23	680.17	wash water in						
	23a	1,170.19							
CB4	23a	1,170.19		from WB3 to CB4		1,170	1,159	(11)	-1.0%
	24	774.30			liquid out of CB4				
	25	385		CB4 to dryer					
DRYER	25	384.71		CB4 to dryer		385	385	-	
	26	82.58		solid for MSU team					
	26a	302			total liquid removed in drying				

The dried fermentation residue (stream # 26) was further ground, using a regular coffee grinder. The ground sample was sieved and the material that passed through a sieve with 42 mesh screen and retained on a sieve with 60 mesh screen was provided to the MSU team for extraction of micro/nano cellulosic fiber.

The detail of the micro/nano cellulosic fiber work is provided in Appendix C.

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## **Appendix A**

**Andritz letter**

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3rd August 2009

David Senyk  
Vice President Engineering  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

Subject: Letter of Confirmation

Dear David:

ANDRITZ is one of the leading suppliers of pulping processes and equipment for all kinds of wood and annual fibers. ANDRITZ is a global company with over 13,000 employees with offices and manufacturing facilities worldwide. ANDRITZ has been active in providing pressurized reactors through our pulp and paper divisions for both wood chips as well as non-woody feedstocks (e.g. bagasse). ANDRITZ has recently intensified their activities in biofuels and biochemicals areas. In the biochemical area this has primarily been with pre-treatment systems (e.g. pre-treatment systems for steam explosion, advanced steam explosion, and dilute acid hydrolysis). In the thermochemical area ANDRITZ provides gasification systems, biomass boilers, etc.

ANDRITZ provides small, medium and very large equipment for traditional pulping applications that is now being utilized for various applications in biofuel / biomass handling. ANDRITZ has also been building smaller pilot and demo systems for the last 25 years (about 100 units and systems in the last 25 years) primarily for the pulp and paper or MDF industries. ANDRITZ also provides equipment for processing feed stocks for food additives, special material recycle systems, and other industries with both wood and non-wood raw materials (forest and agricultural residues, bagasse, leather, plastic residues, fruit baskets, etc)

In the field of biomass processing we supply plant segments and equipment for a wide diversity of raw materials, including:

- Wood
- Straw
- Plastics
- Paper/labels
- Paper pulp
- Sewage sludge
- Industrial waste
- Chemicals
- Municipal waste
- Stabilizers
- Minerals
- Fertilizers

**ANDRITZ LTÉE / LTD.**

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Tel. (514) 631-7700  
Fax (514) 631-3995

In February of 2009 ANDRITZ prepared a technical and commercial budget proposal for a pilot scale biomass AFEX pretreatment system for MBI of Lansing, Michigan. The proposal covered the design and supply of equipment for an approximate 100 kg/hr pretreatment system (pressure feeder, mixing conveyor, horizontal reactor and discharge device) based on corn stover. ANDRITZ is familiar with the AFEX process conditions (temperature, pressure, and residence time) and is confident the equipment specified in our proposal is capable meeting those conditions with either corn stover or wheat straw as the feedstock. ANDRITZ has repeatedly shown this equipment to be capable of meeting similar process conditions with both wheat straw and corn stover in the proposed equipment as well as smaller and larger scale units.

Sincerely yours,



Serge G. Gendreau  
Vice President  
Mechanical Pulping Division  
Andritz Ltd.

**ANDRITZ LTÉE / LTD.**

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## **Appendix B**

**DOE proposal**

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## **Demonstration of an AFEX Integrated Biorefinery at Pilot Scale: Project Summary**

MBI International (MBI) is leading a world-class team of industry, university, and federal laboratory partners to design, construct, and operate an innovative, integrated biorefinery to produce ethanol from corn stover at 1 dry tonne/day (TPD) pilot scale. This team, which includes the National Renewable Energy Laboratory (NREL), Michigan State University (MSU), Novozymes North America, Inc., Vermeer Corporation, ICM Inc., The Andersons, and Airgas, Inc., brings years of experience in design, engineering and construction, feedstocks, biomass processing, enzymatic hydrolysis, microbial strain development, fermentation, and commercialization of biobased products. The MBI team brings a significant portfolio of intellectual property to support the technology, and key technical innovators are integral members of the team. Dr. Bernie Steele, of MBI, will be the Project Director.

This Integrated Biorefinery is underpinned by the Ammonia Fiber Expansion (AFEX) treatment process in which biomass, water and concentrated ammonia are mixed and subjected to elevated temperature and pressure for a brief period. AFEX treatment results in reduced recalcitrance of biomass to subsequent enzymatic hydrolysis, and more effective conversion of resulting sugars to fermentation products. AFEX is a “dry-biomass-in-and-out” process in which the sugars are retained in their polymeric form without any prehydrolysis or partial sugar solubilization. Consequently, AFEX-treated biomass is a *stable intermediate*, one that can be densified, stored, transported and integrated with subsequent bioconversion steps. The unique properties of AFEX-treated biomass give rise to inherent operational flexibility for the AFEX Integrated Biorefinery, addressing supply-chain-logistics challenges, and facilitating broad commercialization and deployment.

The major goals of the pilot-scale AFEX Integrated Biorefinery are to: 1) Operate a continuous AFEX treatment process with ammonia recovery and reuse at 1 TPD; 2) Integrate the AFEX-treated material with high-solids enzyme hydrolysis and mixed sugar co-fermentation steps to demonstrate ethanol production from corn stover at the 1 TPD scale; 3) Evaluate the fermentation residues and determine their value as fuel for steam and electrical generation and 4) Collect performance data and mass and energy balances from these operations and incorporate them into techno-economic models for a commercial AFEX Integrated Biorefinery.

During the proposed project we will 1) procure and pre-process corn stover; 2) treat corn stover with a continuous AFEX process at pilot-scale; 3) recover and reuse the ammonia in the AFEX process; 4) densify the AFEX-treated corn stover to demonstrate storage and transport advantages; 5) perform high-solids enzyme hydrolysis with specifically selected enzymes; 6) ferment both C5 and C6 sugars to ethanol using a recombinant microorganism; and 7) recover the ethanol. The project will include the design and construction of an expansion to MBI’s pilot facility in Lansing, Michigan, to house the AFEX process equipment. Bioconversion to ethanol will be conducted at NREL’s newly expanded pilot facility in Golden, Colorado. The work plan will be completed in an aggressive but realistic time frame of 44 months with a total budget of \$23.5 million. A path to commercialization is outlined in the submission, with the first commercial plant targeted to go online in 2015.

**Expected Benefits:** The envisioned commercial AFEX Integrated Biorefinery produces ethanol in a cost-effective manner in line with DOE’s targets. Life Cycle Analysis shows the potential to reduce greenhouse gas emissions by 65%. Following broad deployment, Petroleum Displacement Analysis shows the potential for AFEX Integrated Biorefineries to displace approximately 120 million barrels of oil/annum by 2030. The project is expected to create or retain more than 100 jobs. There is also a significant opportunity for job creation based on successful commercial deployment of AFEX Integrated Biorefineries, for which the proposed project is a critical enabling step. If cellulosic ethanol produces 16 billion gallons of renewable fuels by 2022 and employment is similar to the current grain ethanol industry, the creation of 535,000 new jobs in cellulosic fuels is possible.



# Demonstration of an AFEX Integrated Biorefinery at Pilot Scale

## PROJECT NARRATIVE

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#### 1.0 INTRODUCTION

MBI International (MBI) is leading a world-class team of industry, university, and federal laboratory partners to design, construct, and operate an innovative, integrated biorefinery to produce ethanol from corn stover at 1 dry tonne/day (TPD) pilot scale. This team brings together the required knowledge, experience, and facilities to meet the broad range of requirements set forth in DE-FOA-0000096 to successfully demonstrate Integrated Biorefinery Operations at pilot scale. The proposed pilot plant operations and the pathway to commercialization are designed to address three key criteria for sustainable biofuels: (i) cost effectiveness of production (ii) scalability to meet the petroleum displacement objectives in the renewable fuels standards and (iii) reduction in greenhouse gas emissions relative to fossil fuels. Our proposal for an innovative, integrated biorefinery is underpinned by the Ammonia Fiber Expansion (AFEX) treatment process.

AFEX is a biomass treatment process in which biomass, water and concentrated ammonia are mixed in a specified ratio, and maintained at a specified temperature and pressure for a specified time period. AFEX treatment causes significant physical and chemical changes in the biomass, resulting in reduced recalcitrance to enzymatic hydrolysis, and more effective conversion of sugars to fermentation products. The applicability of AFEX to a broad range of agricultural feedstocks has been described in the literature (Alizadeh et al., 2005, Teymouri et al., 2004, Teymouri et al., 2005a, Murnen et al., 2007).

AFEX is fundamentally different from “pretreatment” as defined by the U.S. Department of Energy (DOE). The DOE definition of pretreatment is intimately tied to the concept of “prehydrolysis” of sugars from the biomass, as described in the March 2008 DOE Biomass Multi-year Program Plan document (page 3-31): *“In this [pretreatment/pre-hydrolysis] step, the biomass feedstock undergoes a thermochemical process to break down the hemicellulose fraction of the feedstock into a mixture of soluble five-carbon sugars...and soluble six-carbon sugars. This partial solubilization makes the remaining solid cellulose fraction more accessible for enzyme saccharification later in the process.”*

Unlike most pretreatments, AFEX retains the hemicellulose and cellulose sugars in their polymeric form in the treated biomass. AFEX causes physical and chemical changes in the biomass, but there is no pre-hydrolysis of the hemicellulose fraction nor conversion to monomeric sugars during the

process. No sugar-bearing liquid stream is produced during the AFEX process, as is seen in steam or dilute-acid pretreatment processes. Consequently, unlike pretreated biomass, AFEX-treated biomass may be considered a stable intermediate, one that can be stored, transported and integrated with subsequent processing steps.

The DOE-supported Biomass Refining Consortium for Applied Fundamentals and Innovation (CAFI) studies conclude that AFEX is effective and economical. Following extensive comparative studies at laboratory bench-scale, the CAFI group concluded that: 1) AFEX is an effective biomass treatment method to enhance enzymatic hydrolysis of corn stover to produce clean fermentable sugars, and 2) the estimated cost of commercial production of ethanol using AFEX is competitive with other technologies (Wyman et al., 2005, Eggeman and Elander, 2005).

While AFEX is a promising biomass treatment technology, studies to date have been conducted at laboratory bench-scale in batch-mode reactor systems. The next steps in realizing the commercial potential of AFEX are for MBI and its partners to demonstrate an integrated biorefinery based on the AFEX process. This AFEX Integrated Biorefinery includes the steps of (a) feedstock procurement and processing (b) AFEX treatment operating with continuous processing and ammonia recycle and (c) enzymatic hydrolysis, fermentation and ethanol recovery.

This proposal outlines our plans to demonstrate a scalable AFEX process, integrate AFEX with upstream and downstream processes to demonstrate an integrated biorefinery and define a pathway for rapid commercial deployment of AFEX Integrated Biorefineries.

## 2.0 PROJECT OVERVIEW

A flow chart of both a conventional and an AFEX Integrated Biorefinery is shown in Figure 1. The first step in both processes is to grow, harvest, and preprocess the biomass into a stable intermediate – harvested biomass – that can be stored and transported as needed for subsequent steps. Next, in a conventional model, the biomass is pretreated, conditioned, enzymatically hydrolyzed, and fermented, after which ethanol is recovered. Note that no stable intermediate is produced between pre-treatment and the subsequent steps. This means that pretreatment must occur at the same location as all subsequent steps, and processing must be followed through to the final product, ethanol.

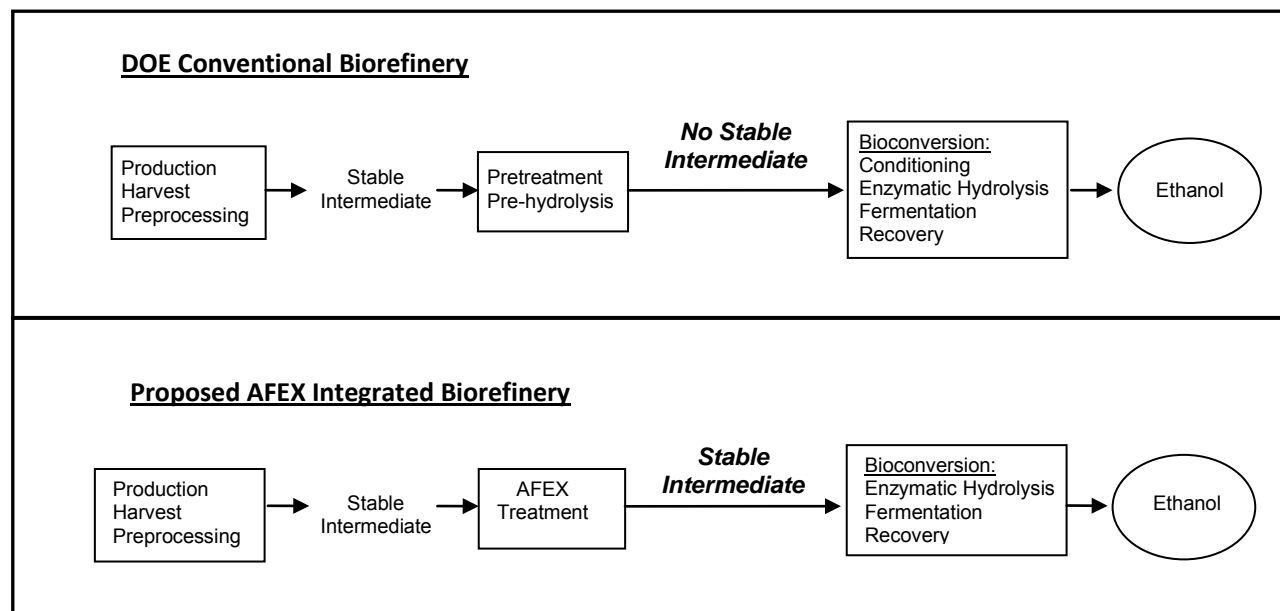


Figure 1. Integrated Biorefinery concepts

For the AFEX Integrated Biorefinery, the preprocessed biomass is subjected to AFEX treatment, which prepares the biomass for bioconversion. Since AFEX is a “dry biomass in - dry biomass out” process, no initial pre-hydrolysis occurs and the resulting AFEX-treated biomass is in a stable intermediate form that can be stored and transported. AFEX-treated biomass does not require subsequent conditioning; therefore, final bioconversion steps are simply enzymatic hydrolysis, fermentation, and recovery.

An AFEX Integrated Biorefinery has inherent flexibility with respect to where the various processes can be located. For example: 1) in a centralized option, AFEX treatment is co-located with the Bioconversion Facility; and 2) in a de-centralized option, AFEX treatment would be located separately from the Bioconversion Facility, with multiple AFEX treatment centers serving a given Bioconversion Facility. We plan to capitalize on this flexibility during the pilot plant and subsequent demonstration and commercial phases. For the proposed pilot plant, we will de-centralize the stages and capitalize on our partners’ existing infrastructure.

An AFEX Integrated Biorefinery is integrated in the following ways:

- 1) It produces ethanol as the primary product from 1 TPD of biomass through the entire process.
- 2) It links each process stage to the next by the flow of a stable intermediate.
- 3) It maintains integrity by restricting energy and recycle streams within a given process stage unless two stages are co-located. The only material transferred from one process stage to the next is a stable intermediate.

#### **Advantages of an AFEX Integrated Biorefinery**

An AFEX Integrated Biorefinery has compelling advantages over the Conventional Biorefinery shown in Figure 1. These advantages arise from the underlying features of AFEX and the properties of the stable AFEX-treated intermediate. Along with these advantages, AFEX treatment does come with the costs associated with ammonia handling, recovery and reuse. These costs are not borne by steam or acid pretreatment, though these pretreatments do bear other costs such as specialized materials of construction, conditioning of pretreated biomass, and waste handling. In any case, the costs of any treatment technology must be viewed in balance with the benefits conferred by the technology, and such cost/benefit analyses are best done at the integrated systems level. Table 1 below summarizes the underlying features of AFEX, the systems benefits arising from these features, and overall outcomes for the proposed AFEX Integrated Biorefinery.

The benefits summarized above are important from a commercial perspective since they address three key criteria for sustainable biofuels -- cost effectiveness, scalability, and reduction in greenhouse gas emissions. The AFEX stable intermediate can be used as a feedstock for other bio-based products as well as animal feed. A product that can be sold into several markets is less vulnerable to the volatility of a single market. This feature reduces risk and could enhance the prospects for rapid and broad commercial deployment.

#### **Innovations of an AFEX Integrated Biorefinery**

In order to realize the significant economic and logistic advantages described above, we propose to demonstrate a scalable AFEX process. Scalability requires the transformation of the batch process into a continuous AFEX process. We propose to then integrate the continuous AFEX process with upstream biomass production/preprocessing steps and downstream bioconversion steps. These combined operations will demonstrate a scalable AFEX Integrated Biorefinery at a 1 TPD pilot scale. Key innovations required to demonstrate a scalable AFEX Integrated Biorefinery include:

- 1) Design and operation of a continuous AFEX biomass treatment process heretofore practiced only in batch mode
- 2) Incorporation of ammonia recovery and reuse in the continuous AFEX process
- 3) Densification of AFEX-treated biomass for storage and transportation efficiency

- 4) Integration of high-solids enzymatic hydrolysis and mixed-sugar co-fermentation steps with AFEX-treated stover at pilot scale
- 5) Incorporation of enzymes selected specifically for efficient hydrolysis of AFEX-treated corn stover

The scalable AFEX Integrated Biorefinery featuring these key innovations will represent a breakthrough technology. The features listed in Table 1 are all examples of differentiated, innovative benefits that will arise from commercialization of this breakthrough technology.

**Table 1. Advantages of an AFEX Integrated Biorefinery**

<u>Feature</u>	<u>Systems Benefit</u>	<u>Overall Outcomes</u>
AFEX can be linked with upstream and downstream steps via stable intermediates	High level of overall process integration from biomass to ethanol	Performs favorably in terms of cost and reduced greenhouse gas emissions
AFEX-treated biomass can be stored and transported	Centralized and decentralized options for integrated biorefinery operation	Flexible deployment of commercial biorefineries in varied geographical areas
Properties of AFEX-treated biomass facilitate densification	Efficient storage and transportation between stages utilizing existing infrastructure	Improves supply-chain logistics
AFEX is effective on multiple agricultural feedstocks	Multiple feedstocks can be processed in a given integrated biorefinery	Mitigates vulnerability to supply chain disruption and facilitates year-round operation
AFEX treatment does not produce high levels of metabolic inhibitors	AFEX treated biomass is compatible with fermentation using diverse organisms	Speeds acceptance of next generation organisms
AFEX treated biomass does not require neutralization, washing or conditioning	AFEX treated biomass can be used as feedstock for a variety of bio-based products	AFEX infrastructure can support both fuels and bio-based chemicals
AFEX-treated biomass can be digested by ruminants	AFEX-treated biomass can be used as animal feed	Addresses food versus fuel tradeoff and land use issues; Reduces market vulnerability

### 3.0 PROJECT OBJECTIVES

The major goals and aims of the pilot-scale AFEX Integrated Biorefinery are outlined below.

#### Goals

- 1) Operate a continuous AFEX treatment process with ammonia recovery and reuse at 1 TPD
- 2) Integrate the AFEX treated material with high-solids enzyme hydrolysis and mixed sugar co-fermentation steps to demonstrate ethanol production from corn stover at the one TPD scale
- 3) Evaluate the fermentation residues and determine their value
- 4) Collect performance data and mass and energy balances from these operations and incorporate them into techno-economic models for a commercial AFEX Integrated Biorefinery

#### Critical Success Factors (CSFs)

Prior work at MBI and MSU has addressed several technical barriers through bench-scale experiments. As explained in more detail in Section 2 of the Project Execution Plan (PEP), bench-scale batch experiments have demonstrated: 1) 94% of theoretical yield of fermentable sugars from AFEX-

treated corn stover; 2) 98% recovery of ammonia from AFEX-treated corn stover; and 3) 5% ethanol titer in high-solids enzymatic hydrolysis and fermentation from AFEX-treated corn stover. These data demonstrate that our AFEX Integrated Biorefinery has significant commercial potential and is ready for validation at pilot scale.

Barriers that will be addressed in the proposed project include (i) uncertainty in the production cost of ethanol in the AFEX Integrated Biorefinery, (ii) viability of operating a continuous AFEX reactor and ammonia catalyst recovery system, (iii) uncertainty in fermentation and recovery efficiencies and (iv) uncertainty in the heating value of fermentation residues.

The factors critical to successful completion of this project are:

1. Complete techno-economic model of a commercial AFEX Integrated Biorefinery, and determine the minimum selling price and GHG emissions for ethanol produced at a commercial facility, using mass and energy flows determined in pilot-scale performance
2. Demonstrate continuous operation of an AFEX reactor with ammonia recycle at pilot scale
3. Integrate high-solids enzymatic hydrolysis and mixed-sugar co-fermentation steps with AFEX-treated stover at pilot scale
4. Achieve the critical factors above and complete all tasks within the scope, budget, and schedule shown in the WBS

### **Value Proposition**

Our proposal meets Energy Efficiency and Renewable Energy performance objectives as outlined by the DOE Biomass Multi Year Program Plan (2008): 1) to reduce dependence on imported oil; 2) to promote the use of diverse, domestic and sustainable energy resources 3) establish a domestic bioindustry; and 4) reduce carbon emissions from energy production. We have the necessary bench-scale technical and economic performance data that validate readiness for scale up to pilot scale. Our AFEX Integrated Biorefinery will demonstrate the production of ethanol, a primary liquid transportation fuel that supports the advanced biofuels portion of the Energy Independence and Security Act of 2007 and the Renewable Fuel Standards of 2007. Our integrated process employs a unique treatment method, minimizes waste, reduces green house gas emissions by 65%, is highly scalable, utilizes an available feedstock and has the flexibility to operate AFEX treatment at locations other than a centralized biorefinery. To support DOE's goals, our team will proceed rapidly through to demonstration-scale and commercialization. We have a sound business and technology strategy to deploy and/or license and market the technology commercially.

Our team proposes an innovative approach to the integrated biorefinery that is enabled by the AFEX treatment process. This approach addresses several barriers to cost effective production of ethanol from lignocellulosic biomass including: 1) recalcitrance of lignocellulosic biomass to enzymatic hydrolysis; 2) catalyst recovery and recycle; 3) high solids loading of treated materials for enzyme hydrolysis and fermentation; 4) enzyme cost; 5) compatibility of biomass sugar stream with fermentation; and 6) opportunity for improvement in supply chain issues.

MBI is a leader in delivering commercially-viable product and process solutions to address the emerging need for new bio-based products that combine environmentally friendly attributes, sustainable production processes, and renewable raw-material sources. MBI applies multidisciplinary expertise, modern laboratories and a robust, flexible de-risking approach. We utilize pilot-plant facilities to accelerate the development and scale-up of bio-based technologies and we partner with both universities and end-user companies to bridge the gap between early innovations and commercial applications. For this proposal, we have assembled a team of partners that, along with MBI personnel, bring years of experience in design, engineering and construction, biomass processing, enzymatic hydrolysis, strain development and fermentation. This MBI team also brings a significant portfolio of intellectual property to support the technology to be demonstrated and the inventors of the technology

are integral members of the team. We have a unique framework of collaborators that have the experience and full spectrum of capabilities for successful project implementation.

The major team members are:

**MBI** - MBI International has a successful 27-year history of bringing renewable products from academic research to the commercial market. One example is polylactic acid (PLA). Engineering, scale-up and applications research for production of lactic acid and PLA biodegradable plastics technology were conducted at MBI and MSU with collaboration and support of Cargill Inc., one of the world's largest agribusinesses. Today, this biodegradable polymer is broadly used around the world in both plastics and fibers in clothing and carpets, containers for food and garbage bags, car parts, etc. Another example, Evercorn, Inc., was a successful joint venture between an MBI subsidiary and Japan Corn Starch (one of Japan's leading starch-based industrial products companies) to develop a family of polymer resins that are processed into films and moldable products for disposable use applications. These polymers are strong, water-resistant thermoplastics used in disposable cutlery, plastic containers, and paper coatings. Evercorn's biodegradable products were featured at the Nagano Olympic Games (1998).

**Vermeer** – Vermeer Corporation manufactures agricultural, construction, environmental, and industrial equipment in Pella, Iowa. From modest beginnings, quality product innovations and demand has allowed the company to expand its offerings to more than 60 countries. Today Vermeer consists of eight manufacturing plants spanning some 110 acres that occupy more than 1.5 million square feet of manufacturing space in Pella, Iowa. Vermeer is recognized as a global leader in forage, tree care, wood-waste processing, composting, compact and underground installation equipment. Vermeer currently employs over 2000 people and is interested in the use and development of its machinery in the cellulosic biofuels industry.

**MSU** - Michigan State University pioneered the concept of the Land Grant University and has the fundamental infrastructure necessary to establish the foundation for bioeconomy development in Michigan. This includes laboratories and core facilities for instrumentation, computation and modeling; field research facilities across the state; funding for creative new research projects; and pilot and scale-up facilities for unique commercialization expertise through MBI International, a firm with proven success in bringing biotechnology ideas to the market. MSU also has strongly developed links with other research universities and private sector partners, including established relationships with all major agricultural input providers, select biotechnology firms and key manufacturing industries.

**NREL** - The National Bioenergy Center (NBC) of the National Renewable Energy Laboratory (NREL) has led the nation's efforts in the R&D of bioprocess conversion technology for lignocellulosic ethanol and other fuels and chemicals from biomass for over 30 years. In addition to its world-class bioprocessing laboratories and pilot plant facilities, the NBC is home to over 100 technical and support staff who are focused on R&D of biochemical and thermochemical conversion process for lignocellulosic biomass. Many researchers in the NBC have over 15 years of experience in the development of such processes, including key personnel who will be leading and contributing to the MBI-led project team. Personnel in the NBC lead and conduct core research tasks for DOE's Office of the Biomass Program (OBP), including several activities with multi-million dollar annual budgets that span the entire range of biochemical conversion technologies, including biomass pretreatment, enzymatic hydrolysis, fermentation, bioprocess integration and scale-up, chemical analysis, and techno-economic analysis. The activities represent the core R&D of DOE's OBP, and NBC researchers are renowned world-wide as experts in all conversion technology aspects, with an extensive patent and publication portfolio.

Additionally, NBC staff members have extensive experience in leading and conducting R&D programs in collaboration with numerous industrial partners to further the deployment of biomass conversion bioprocesses. In recent years, key industrial collaboration projects that the NBC has provided a major technical role included those with DuPont, Abengoa, POET, ADM,

NatureWorks/Cargill-Dow, New Energy Company of Indiana, and Amoco, among others. The NBC staff is proficient in all aspects of managing and conducting such collaborations and has extensively utilized the NBC laboratory and pilot plant facilities in these projects.

**Novozymes** – Novozymes is a world leader in enzyme development, production and sales and has lead the way in development of next generation enzymes for conversion of cellulosic feedstocks to biofuels and chemicals. Through prior work with DOE they have dramatically increased the efficiency and lowered the cost of these enzymes. Novozymes is currently investing \$160-200 million in new facilities in Blair, NE to meet demand for enzymes for the production of first and second generation bioethanol. The new facility will be fully operational in approximately 2010.

**Airgas Specialty Products** – ASP is a leading distributor of anhydrous ammonia and related services in the United States. The company operates an extensive network of 24 ammonia distribution, field service and office locations. The company is a primary supplier of ammonia to the fast-growing market segment of power plants, oil refineries and other manufacturing facilities nationwide who require ammonia for nitrogen oxide removal. ASP also serves its customers with risk management programs, safety training and equipment and related ammonia quality services.

**ICM** - ICM is the nation's leading provider of ethanol process design and engineering, with 78 dry-mill fuel ethanol plants operating in the United States and Canada that use ICM ethanol process technology. ICM-designed plants consistently perform 24 hours per day, at least 353 days per year for a 96% runtime – the highest performance measure in the industry. ICM's market success, an approximate 50% market share in current ethanol production, results from the fact that ICM process technology simply runs better, faster, cheaper and with less downtime than competing technologies. ICM employs 30 full-time engineers in its various departments, including process chemical engineers, mechanical engineers, electrical engineers, and civil engineers. ICM also designs, engineers, and installs a proprietary distributive control system (DCS) for ethanol plants that use ICM process technology.

**The Andersons** - The Andersons is a publicly traded company with 3000 employees and diverse interests that include agribusinesses such as grain and plant nutrients as well as railcar leasing and repair, industrial products formulation, turf products, retailing and most recently, ethanol operations. The Grain Division operates grain terminals in Ohio, Michigan, Indiana, and Illinois with storage capacity of nearly 90 million bushels. The division trades and merchandises more than 250 million bushels of grain in the Eastern Corn Belt and Canada. The Ethanol Division operates three plants; a 55 million gallon/yr plant in Albion, Michigan and two 110 million gallon/yr plants with partner Marathon Petroleum Company.

### **Readiness to Proceed**

MBI has assembled a leading group of partners to work with the innovative AFEX Integrated Biorefinery. The concept for commercialization offers a unique method of improving the supply chain management and economics that have burdened many plans for cellulosic biorefineries. The proposed plan makes use of each partner's unique expertise and facilities. All team members are budget and milestone driven with experienced performance in delivering project results on time and on budget. In addition, significant preliminary work has been done so that the project can move rapidly through the preliminary stage and on to construction.

MBI and its team partners are ready to proceed with the proposed pilot scale AFEX Integrated Biorefinery. Facilities at MBI and NREL are currently permitted for proposed operations and a quantitative process design has been completed. In addition most major equipment vendors for the AFEX treatment plant have been contacted and recent equipment quotations are available.

#### **4.0 PROJECT DESCRIPTION**

This section describes the background, tasks, activities, key decisions, the project management processes and the associated budgets that will be undertaken to address the project goals described in Section 3 of this Narrative.

##### **Project Background**

The AFEX Integrated Biorefinery is ready to transition to pilot scale. More than 20 years of bench-scale studies on AFEX treatment of biomass have been conducted and may be summarized as follows: (i) AFEX biomass treatment technology has been demonstrated to work on corn stover; (ii) AFEX-treated biomass has been shown to be amenable to densification (iii) AFEX-treated biomass has been shown to be stable upon storage; (iv) AFEX-treated corn stover has been shown to be amenable to saccharification with commercially available hydrolytic enzymes to yield a mix of C5 and C6 sugars; (v) AFEX-treated biomass has been shown to be compatible with saccharification operations at high solids loading; (vi) C5/C6 mixed sugar stream arising from AFEX treated biomass has shown to be fermented to ethanol by a variety of organisms, including *Zymomonas mobilis*; (vii) It has been shown that AFEX-treated biomass does not produce significant levels of microbial growth and fermentation inhibitors; and (viii) The recovery of up to 98% of the ammonia from AFEX-treated biomass has been demonstrated. Detailed background and data related to the above are provided in Section 2 of the PEP.

We have developed a Pro forma projection for an envisioned commercial AFEX Integrated Biorefinery to become operational in 2015 (see Proforma.xls). The assumptions used in this Pro forma are consistent with published technical reports from NREL (Aden, 2008), CAFI (Eggeman and Elander, 2005), MBI (Campbell et al., 2008; Campbell et al., 2009), and MSU (Laureano-Perz et al., 2005; Teymouri et al., 2004; Teymouri et al., 2005a; Teymouri et al., 2005b). The envisioned commercial AFEX Integrated Biorefinery will produce 120 million gallons of ethanol/yr. A significant component of the projection is a residue-combustion plant that produces all heat and power for the facility and sells power to the grid, generating \$8.2 million in income.

Using the value of fuel, the financing terms, and the inflation factor provided in the Pro forma forms, the envisioned commercial plant in 2015 produces ethanol for \$2.19/ gallon with a 21.6% return on Total Project Investment (TPI). The minimum ethanol selling price (MESP) to meet a 9.02% return on TPI is \$1.58/gallon without financing. This MESP is equivalent to the \$1.49/gallon projection (for 2012, also without financing) made by NREL at the 2009 Symposium for Biotechnology for Fuels and Chemicals (Humbird et al., 2009) for the dilute acid process when adjusted for inflation at 2.4%/yr to the year 2015. The AFEX Integrated Biorefinery thus provides a cost effective way to produce cellulosic ethanol.

The expected reduction in greenhouse gas emissions for ethanol produced from corn stover in a commercial AFEX Integrated Biorefinery is 65% and the expected reduction of fossil fuel usage is 73%. Further details on these projections are provided in the Life-Cycle GHG Emission Reduction Data (LCA\_GHG.xls). The expected petroleum displacement for a commercial AFEX Integrated Biorefinery is 400,000 barrels per year. Further details on this projection are provided in the Petroleum Displacement Analysis (Petro.doc).

##### **Tasks/Activities**

This section describes the tasks and activities that will be undertaken to meet the project goals. We are proposing an innovative approach to the pilot-scale AFEX Integrated Biorefinery that capitalizes on the stable intermediates produced in the process, namely harvested corn stover and AFEX-treated corn stover (Figure 1 in Section 2 of this Narrative). These stable intermediates allow the operations to be de-centralized at different partners' locations while maintaining process integration as described in Section 2 of this Narrative. Our approach leverages partners' existing infrastructure, which in turn maximizes the overall cost-effectiveness of the investments, and more importantly, accelerates timelines. The accelerated timelines make this project more amenable with the timelines required by



the American Recovery and Reinvestment Act of 2009 (Recovery Act), while also addressing the national timelines and goals for replacing petroleum and reducing greenhouse gas emissions.

The proposed project calls for: 1) partner Vermeer, to source and prepare the corn stover (Iowa); 2) AFEX treatment in a 1 TPD AFEX processing plant to be constructed and operated at the MBI facility in Lansing, Michigan; 3) high-solids saccharification and mixed sugar co-fermentation at the newly-expanded NREL pilot plant in Golden, Colorado; and 5) MBI and NREL to analyze the composition and determine the value of the fermentation residue. Data collected from the pilot biorefinery will be incorporated into techno-economic models for commercial plants.

The specific roles of the organizations during the proposed pilot-scale project are summarized below.



Location	Iowa	Michigan	Colorado
Primary Role	Vermeer	MBI	NREL
Secondary Role	MSU	MSU/Airgas	MBI/Novozymes/MSU

**Figure 2. Locations and roles of organizations during proposed pilot-scale project.**

The detailed Work Breakdown Structure (WBS) can be seen in Section 1 of the Project Management Plan (PMP). There are three Budget Periods of activity throughout the Pilot Scale project; Budget Period 1 includes the design, equipment selection, R&D, NEPA determination, Risk Mitigation Plan, final site and construction plan, and construction permits; Budget Period 2 includes the AFEX building construction, equipment installation, shakedown trials, and demonstration (capstone) runs; Budget Period 3 includes on-going pilot-scale operations of the AFEX Integrated Biorefinery, data reporting, and economic modeling.

The R&D effort is aimed at mitigating specific risks as detailed in the Risk Management Plan (Section 4.0 of the PMP), and includes work focused on ammonia recovery and recycle, enzyme selection and loading, and the challenges of pilot scale high solids loading of AFEX-treated corn stover into the hydrolysis and fermentation modules. The information from this work will be integrated into a final design for the integrated biorefinery.

With respect to AFEX equipment design and construction, we are in an advanced state of readiness to proceed because of historical investments made prior to the current FOA. In 2005 MBI commissioned a preliminary design for a 35 TPD AFEX pilot plant from Dick Engineering (Toronto, Ontario). Dick Engineering recently helped MBI revise and update this design, with cost estimates, for 1 TPD scale. Process equipment, piping and electrical costs were included. Budget quotations for major equipment were obtained. We have visited and had numerous discussions with potential vendors of primary equipment related to AFEX processing and ammonia recovery and have preliminary quotations for this equipment. A preliminary environmental assessment has not revealed any impediments to construction and operation of the proposed pilot plant. All facilities are fully permitted for the proposed operations, including storage and handling of anhydrous ammonia. Only conventional building

construction permits will be needed during the course of the project. This should enable our team to progress swiftly through final NEPA approval for the project.

The equipment and facility design phase is expected to take 4-6 months. It is expected that initial procurement of primary equipment for the AFEX reactor can begin within ten months of the project start and will continue through most of the construction phase. It is necessary to begin procurement as early as possible due to the long lead times (estimates supplied by vendors) of the customized equipment required for the pilot plant. The rest of the process design is dependent on the specific equipment selected for the AFEX reactor. The construction and equipment installation phase of the project is expected to take approximately 12 months and will commence when sufficient areas of design are completed for that portion of the project and DOE has approved start on Budget Period 2.

To house the AFEX treatment and ammonia recovery equipment, MBI plans to make an addition to its facility. The addition will be built adjacent to the existing facility in order to use the existing infrastructure. Steam, water, waste disposal, and electrical power distribution capacity already exists that is sufficient to supply the AFEX treatment pilot plant. The addition will match the original construction and appearance of the existing building. Interior architectural details will match MBI's existing building including the masonry walls. One side of the building will have a receiving and storage area for biomass feedstock with direct access to the processing equipment. The building will also feature an electrical/utilities room, control room, and a laboratory area. An employee workstation and meeting area will be located in the building. The building cost estimate was prepared by EPS (South Bend, Indiana), an engineering design firm with 14 years experience in the biotech and energy industries.

Equipment shakedown refers to the process of troubleshooting to achieve functional operation of all process equipment. Due to the uniqueness of the AFEX process, in particular the use of ammonia recovery and recycle, it is expected that the shakedown phase of the project will require approximately 9 months for AFEX and densification operations and 9 months for the hydrolysis and fermentation operations. All reasonable efforts will be made to keep this phase to a minimum while maintaining appropriate quality assurance and safety standards.

Capstone runs refer to a planned campaign of operations of the proposed biorefinery to successfully demonstrate the feasibility of the technology. Approximately twelve months have been allocated for the capstone runs to demonstrate the functionality, reliability and performance of the AFEX Integrated Biorefinery. This is based on the concept of three campaigns similar to industry standards for scale up of commercial fermentations.

Techno-Economic models will include detailed ASPEN PLUS process models with ASPEN ENCARTA costing modules. These models will contain all unit operations, mass and energy balances, and feed, product, co-product and internal streams necessary for proposed commercial operations. These ASPEN models will be combined with Microsoft Excel models for feedstock preparation and logistics, and business and financial plans to create Pro formas for commercial operations. Models will be created for both centralized and decentralized commercial options to update commercialization plans.

Extended operations will be conducted during Budget Period 3 to collect additional data and generate information for input into the techno-economic models, life-cycle analyses and DOE's State of Technology reports.

### **Schedule, Resource Loaded Plan, and Spend Plan**

As noted above, the full WBS and resource loaded plan can be seen in Section 1 of the PMP. Table 2 below shows a summary of the schedule, major tasks/activities, and spend plan for our pilot plant proposal.

The total pilot plant project cost is \$23.5 MM over 44 months. The first year will include final R&D work, final design work and initial procurement activities at a cost of \$5.4 MM for the year. During

the second year procurement is completed, and construction begins for a cost of \$9.7 MM for the year. In year three, construction is completed, as well as facilities shakedown and final demonstration runs required to meet the goals and objectives of the project for a total of \$6.7 MM. During the fourth year MBI will continue to operate the pilot plant for approximately 8 months to gather data crucial to design and construction of a demonstration scale plant (\$1.7 MM).

### **Key Decision Points and Project Management**

MBI's current project management practice is consistent with DOE O 413.3A and addresses such elements as strategic planning, roles and responsibilities, work planning and control, fact-based decision making, Stage-Gate reviews, financial planning and control, and reporting. As described in the Project Management Plan, MBI uses a Stage-Gate approach to project management, including go/no-go decision points. Our Stage-Gate process is a method for making disciplined decisions about research and development that leads to focused process and/or product development efforts, and is coordinated with our resource loaded plan. Further details on our approach and how we employ this method to measure progress towards achieving our goals is seen in Section 3.2.2 of the PMP and Section 4.3 of the PEP. There are four Stage-Gate Reviews planned for this project. Expected dates for the Stage-Gate reviews and their expected go/no-go decisions are as follows:

Stage-Gate I – September 2010

- NEPA determination must be complete
- Construction plan must be complete
- Building permits must be in hand

Stage-Gate II – November 2011

- Construction and equipment installation are complete and ready for shakedown

Stage-Gate III – January 2013

- Capstone (demonstration) runs are completed
- Techno-economic models are updated using capstone run data

Stage-Gate IV – July 2013

- Techno-economic models justify a demonstration or commercial scale project

In addition to the above, certain critical milestones in Budget Period 2 are go/no-go decisions. If these milestones are not made on time, some recycling would need to be done and delays could be expected until each was completed. These important milestones and their expected dates of completion are:

1. MBI building construction passes inspection for occupancy – 6/2011
2. Independent Engineer approval of AFEX and Densification equipment at MBI – 12/2011
3. Independent Engineer approval of Hydrolysis and Fermentation equipment at NREL – 8/2012

### **5.0 Path to Commercialization**

MBI and its partners have envisioned a clear and aggressive conceptual path to commercialization, with the first commercial plant coming on line in 2015. We believe that this aggressive timeline is achievable because: (i) the primary product is ethanol, which has established market and distribution channels; (ii) we will target existing grain ethanol facilities to which AFEX units will be added; (iii) AFEX equipment can be based on equipment used in alkaline pulping plants; such processes are routinely operated at > 1000 TPD output; and (iv) the financial returns for the envisioned commercial AFEX Integrated Biorefinery are compelling, and should therefore attract capital.

The first step in this path is the proposed pilot scale project. During the first two years on this path (Figure 3), we will build the proposed pilot plant to demonstrate the AFEX Integrated Biorefinery at 1 TPD (Budget Periods 1 and 2). Upon completion of the pilot plant, we will execute the proposed work to collect the mass and energy balances, Life Cycle Analysis (LCA), Techno-Economic data and Capital and Operating cost data necessary to incorporate in a model for a demonstration/commercial plant

(Budget Periods 2 and 3). These data will help us track and understand the supply chain logistics and potential economic and technical utility of both de-centralized and centralized commercial operating strategies (Section 5 of the Business and Commercialization Plan (BCP). In a decentralized option, the size of a demonstration and commercial facility may be similar. In years three and four of this path, in parallel with pilot plant operations, we will partner with key commercial entities to plan a demonstration/commercial facility. During years four to six of this path, we will execute the demonstration/commercial facility plan with our commercial partners. Such a facility will allow us to validate assumptions based on larger-scale operating data, leading to LCA and techno-economic models necessary to determine expanded commercial implementation. We plan to then broadly expand the deployment of AFEX Integrated Biorefineries through licensing arrangements.

**Table 2. Spend Plan**

Budget Period	Category	2010	2011	2012	2013	Totals
<b>1</b>	R&D	\$1.5				\$1.5
	Design, Bid, Procure	\$0.9				\$0.9
	Lab Equipment	\$0.2				\$0.2
	Model & LCA	\$0.1				\$0.1
	Proj. Mgmt, Risk Mitigation, NEPA, Reporting	\$0.3				\$0.3
	<b>Total</b>	<b>\$3.0</b>				<b>\$3.0</b>
<b>2</b>	Equipment	\$1.6	\$3.9			\$5.5
	Design, Construction & Installation	\$0.6	\$5.2			\$5.8
	Shakedown Capstone Runs			\$5.7		\$5.7
	Model & LCA			\$0.5		\$0.5
	Proj. Mgmt, Risk Mitigation, Reporting	\$0.2	\$0.6	\$0.5		\$1.3
	<b>Total</b>	<b>\$2.4</b>	<b>\$9.7</b>	<b>\$6.7</b>		<b>\$18.8</b>
<b>3</b>	Operation				\$1.5	\$1.5
	Proj. Mgmt., LCA, Modeling, Reporting				\$0.2	\$0.2
	<b>Total</b>				<b>\$1.7</b>	<b>\$1.7</b>
<b>Total Budget</b>		<b>\$5.4</b>	<b>\$9.7</b>	<b>\$6.7</b>	<b>\$1.7</b>	<b>\$23.5</b>

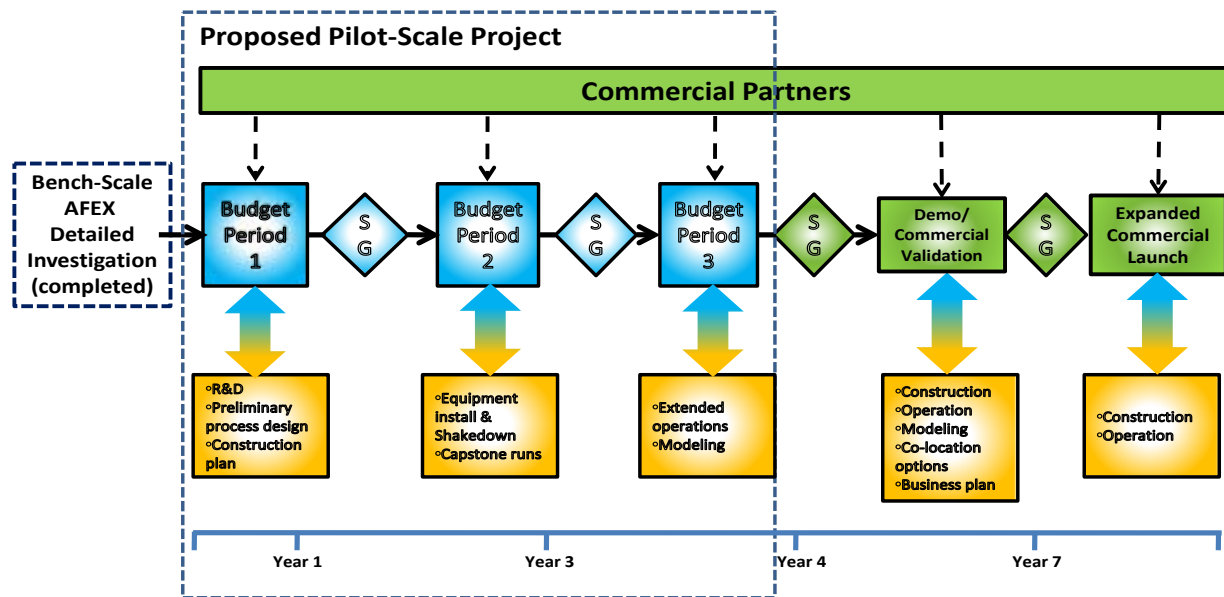


Figure 3. AFEX integrated Biorefinery Path to Commercialization

## 6.0 Outcomes Related to the Recovery Act

This project promotes and enhances the objectives of the American Recovery and Reinvestment Act of 2009 (Recovery Act). The project is positioned to move expeditiously with construction of new facilities. The project will create and retain jobs in the professional, technical, construction and skilled labor sectors. The project also leverages each partner’s existing expertise and facilities, resulting in execution of the work plan in a compressed time frame and thereby accelerating economic recovery.

This project is “shovel ready”. A significant level of preliminary design and planning has been completed for the new continuous AFEX treatment facility to be constructed at MBI. NREL is currently in the process of constructing new expanded pilot facilities for high solids processing and fermentation of biomass feedstocks. This advanced level of planning and construction gives us confidence in the proposed timelines for tasks and completion dates. We plan to complete this project in three years and eight months. This aggressive timeline is consistent with the expectations of the Recovery Act.

A significant number of jobs will be created or retained as a result of this project. These jobs may be classified into three categories: (i) Team MBI personnel involved in directly executing the project; (ii) personnel involved in the design and construction of the pilot-scale AFEX processing facility at MBI; and (iii) personnel involved in the design, fabrication, and installation of the specialized equipment used in the project.

In the first category, we estimate that a total of approximately 56.8 Full Time Equivalents (FTE’s) will be created or retained over a 44-month period at MBI and partners MSU, NREL, Novozymes and Vermeer. These jobs involve technical experts, technicians or pilot plant operating personnel – all of which are highly relevant for the emerging renewable energy sector (“green jobs”).

In the second category, we estimate that the design, equipment installation and construction of the AFEX pilot plant facility in Lansing, Michigan will last 13 months and cost \$2.8 million. Assuming an average fully absorbed rate of \$130,000 per FTE for these jobs, we estimate that 21 FTE’s will be created or retained in this category over the 13-month period.

In the third category, we estimate that the design, fabrication of the AFEX pilot plant equipment will last approximately 10 months and cost \$ 5.2 million, of which 50% will be spent on materials and the remaining on labor. Assuming a fully absorbed rate of \$100,000 per FTE, we estimate that 26 technical

jobs will be created or retained in this category.

Note that many of these jobs will be located in Michigan, the state that has the highest unemployment rate in the nation, and is greatly in need of economic stimulus. The State of Michigan, the Governor and the Economic Development Group see the advantages of a developing alternative energy industry for economic recovery in Michigan. Commercialization of cellulosic fuels in Michigan can make a significant contribution to this economic development plan. Also note that we are underestimating the total number of jobs in all the categories above since we are not able to accurately estimate the number of jobs created or retained in operations such as raw material production, refining, formulation and transportation.

Finally, there is a significant opportunity for job creation and economic recovery based on successful commercial deployment of AFEX Integrated Biorefineries, for which the proposed project is a critical enabling step. According to the Renewable Fuels Association ([www.ethanolrfa.org](http://www.ethanolrfa.org)) the ethanol industry created 154,000 U.S. jobs in 2005 alone, boosting household income by \$5.7 billion. It also contributed about \$3.5 billion in tax revenues at the local, state, and federal levels. If cellulosic ethanol is to produce 16 billion gallons of renewable fuels by in 2022 and employment is similar to the current grain ethanol industry, we could expect the creation of 535,000 new jobs in cellulosic fuels by 2022.

## **7.0 CONCLUSION**

This proposal warrants the support of the DOE because our scalable AFEX Integrated Biorefinery will address three critical issues that face sustained biomass-to-ethanol production: cost effectiveness, scalability, and greenhouse gas emissions reduction.

The AFEX Integrated Biorefinery is a breakthrough technology comprising multiple innovations for improved biomass processing, supply chain logistics, and bioconversion efficiency. The successful demonstration of these innovations will enable the proposed biorefinery technology to proceed rapidly and cost effectively to commercialization.

MBI International has a successful track record of leading technology development collaborations and accelerating the commercialization of bio-based products. MBI will lead a strong team of collaborators from industry, academia and federal laboratories to design, construct, and operate an innovative, integrated biorefinery at pilot scale. The collective knowhow and experience of the team gives the project a high probability of success.

Successful completion of this project will advance the national goals of (i) reducing dependence on imported oil (ii) spurring the creation of a domestic bio-industry and (iii) creating and retaining jobs and promoting economic recovery.

**MICHIGAN STATE**  
**UNIVERSITY**

June 16, 2009

Venkataraman (Bobby) Bringi, Ph.D.  
President/CEO  
MBI International  
3900 Collins Road  
Lansing, MI 48910

Subject: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096:  
Demonstration of Integrated Biorefinery Operations

Dear Dr. Bringi,

I am writing to express my unqualified support for MBI's proposal titled "Demonstration of an AFEX Integrated Biorefinery at Pilot Scale". I am fully aware of the scope of the project, the financial investments involved, and the potential risks and upsides associated with the project. Michigan State University is a willing participating partner in this proposal and has prepared a separate letter of commitment as required.

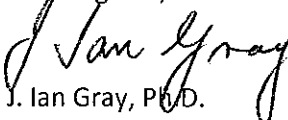
MBI and MSU had in 1998 established a collaboration to develop and de-risk AFEX biomass treatment technology, developed initially by MSU faculty member, Professor Bruce Dale. The current proposal is a logical step to validate the viability of AFEX at pilot scale and to integrate AFEX with the rest of the processing steps to demonstrate an AFEX Integrated Biorefinery at pilot scale. I believe that the proposed AFEX Integrated Biorefinery has the potential to achieve the DOE's goals for the sustainable production of cellulosic biofuels and the reduction of Green House Gas emissions.

MBI and the partners you have assembled are uniquely positioned to meet the challenges represented by the scope of the project, on an aggressive timetable. This team has the substantive experience, intellectual, and financial resources that are required to successfully complete the project within the proposed time and budgets.

The proposed work is complementary to the mission and priorities of Michigan State University. MSU has made a major commitment to the Bioeconomy, as evidenced in part by its participation in the Great Lakes Biotechnology Research Center (GLBRC), funded by the US Department of Energy. I believe that a biomass processing pilot plant, operating at MBI, will greatly enhance current biomass processing programs at the University and within the GLBRC. As a result, MSU and MBI together will be better positioned to accelerate promising biomass-based technologies from early concept to the market.

On behalf of MSU, I am pleased to express my unqualified endorsement of MBI's proposal and would urge DOE's consideration of an award to you and your team.

Best Regards,



J. Ian Gray, Ph.D.  
Vice President for Research and Graduate Studies  
Ex-Officio Member, MBI Board of Directors



OFFICE OF THE  
**VICE PRESIDENT  
FOR RESEARCH  
AND GRADUATE  
STUDIES**

**J. Ian Gray**  
Vice President

Michigan State University  
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517/355-0306  
FAX: 517/432-1171  
[www.msu.edu/unit/vprgs](http://www.msu.edu/unit/vprgs)

June 23, 2009

D. Bernie Steele, Ph.D.  
Director-Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

RE: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096: Demonstration of Integrated Biorefinery Operations

Dear Dr. Steele:

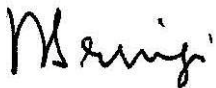
This letter is to confirm MBI International's commitment to participate and lead a world class team to demonstrate a pilot-scale AFEX Integrated Biorefinery in response to the DOE solicitation referenced above. A separate letter has been provided as requested to express senior management buy-in to the project, as well as to demonstrate a financial commitment from MBI's parent organization, the MSU Foundation.

As a leader and participant in this project, MBI staff will play key roles in AFEX Treatment; Enzyme Hydrolysis; Fermentation Scale-Up; Plant Design and Engineering; Plant Operations and Maintenance; NEPA and Environmental Permitting; Techno-Economic Analysis; and Commercialization Plan Development. MBI personnel will also participate in quarterly meetings and StageGate reviews.

Using the MBI's normal accounting practices, we have determined the budget for MBI's portion of the project (does not include \$3,473,313 for the National Renewable Energy Laboratory in Golden, Colorado) to be \$20,014,483, which includes \$634,811 in subawards to Michigan State University, Novozymes and Vermeer Corporation. MBI will contribute \$4,342,741 (approximately 18.5% of the total project cost of \$23,487,796) as cost share in the form of in-kind services.

We look forward to working with the excellent team that MBI has assembled to demonstrate the AFEX Integrated Biorefinery at pilot scale and to ready it for commercialization.

Sincerely,



Venkataraman "Bobby" Bringi, Ph.D.  
President and CEO, MBI International



June 19, 2009

D. Bernie Steele, Ph.D.  
Director, Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910

Subject: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096: Demonstration of Integrated Biorefinery Operations

Dear Bernie,

I am writing to express senior management buy-in for MBI's proposal titled "Demonstration of an AFEX Integrated Biorefinery at Pilot Scale" in response to the DOE announcement referenced above. I am fully aware of the scope of the project, the financial investments involved, and the potential risks and upsides associated with the project. I have made key MBI stakeholders and the MBI Board of Directors fully aware of the strategic, financial and operational issues related to MBI's submission.

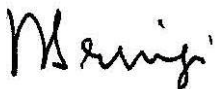
I have obtained the buy-in and support from the MBI Board of Directors and the MSU Foundation. MBI is a wholly owned subsidiary of the MSU Foundation, and will rely on the Foundation for financial support, which in turn will enable MBI to contribute the cost share required for the proposed project. Further, the MSU Foundation is the owner of the current MBI facility, which will be expanded to house the pilot scale AFEX facility. The attached letter from George Benson and Dr. Ronald Goldsberry outlines the MSU Foundation's and the MBI Board of Directors' support for the financial contribution and the facility expansion.

I have obtained the buy-in and support from the MSU Vice President of Research and Graduate Studies, Dr. J. Ian Gray (see Gray letter). MSU is a key player in the MBI team since AFEX was developed initially by MSU faculty member Dr. Bruce Dale, and MSU is the assignee of the associated intellectual property. MBI has an exclusive license to AFEX technology from MSU. I have also obtained the support of the Great Lakes Bioenergy Research Center (GLBRC), in which MSU is a partner. GLBRC is supportive of MBI's proposal since it would provide an avenue for future research innovations to effectively reach the market place.

MBI's strategy is to bridge the gap between bio-based research and commercial application through collaborative de-risking and external alliances. The proposed project is fully consistent with MBI's strategy, and has the potential to address important national goals of energy security and job creation.

On behalf of MBI, I am pleased to express my unqualified endorsement of and buy-in for MBI's proposal. I commend your efforts to date, and wish you and the team the very best of success.

Sincerely,



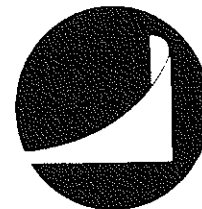
Venkataraman "Bobby" Bringi, Ph.D.  
President and CEO, MBI international  
Member of the MBI Board of Directors

# MICHIGAN STATE UNIVERSITY FOUNDATION

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June 16, 2009

Venkataraman (Bobby) Bringi, Ph.D.  
President/CEO  
MBI International  
3900 Collins Road  
Lansing, MI 48910

Subject: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096:  
Demonstration of Integrated Biorefinery Operations

Dear Bobby,

We are writing to express our unqualified support for MBI's proposal titled "Demonstration of an AFEX Integrated Biorefinery at Pilot Scale". In various briefings and presentations over the past six months, you and your management team have made us fully aware of the scope of the project, the financial investments involved, and the potential risks and upsides associated with the project.

The MSU Foundation ("the Foundation"), a Michigan-based not-for-profit organization with over \$300 million in assets, has a mission to further the interests of Michigan State University. The Foundation acquired MBI in 2005 and has since developed a strategy for MBI to de-risk, scale up and commercialize MSU innovations, thereby accelerating promising technologies from early state to the market. The proposed DOE submission is a logical step to scale up and integrate AFEX technology, and to commercialize it to enable the pressing national goals of fuel security and sufficiency. The successful execution of this project will create and retain highly skilled jobs in Michigan, and would thus also be consistent with the objectives of the American Recovery and Reinvestment Act of 2009.

On behalf of the Foundation and the MBI Board of Directors, we are writing to specifically express our support for the following items: (i) the Foundation stands behind MBI's commitment to provide a total of \$4.4 million in cost share over the period of the proposed project and

Mr. Bobby Bringi  
Page 2  
June 16, 2009

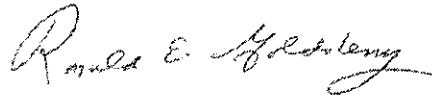
(ii) as the owner of the current MBI facility at 3900 Collins Road in Lansing, the Foundation supports the proposed facility expansion described in the submission.

We commend MBI and its partners for the efforts to date and wish you a successful outcome.

Sincerely,

A handwritten signature in black ink, appearing to read "George Benson", written over a horizontal line. The signature is stylized and somewhat cursive.

George Benson  
Executive Director, MSU Foundation  
Member of the Board of Directors, MBI International

A handwritten signature in black ink, appearing to read "Ronald E. Goldsberry", written over a horizontal line. The signature is cursive and somewhat stylized.

Ronald Goldsberry, Ph.D.  
Member of the Executive Committee and Board of Directors, MSU Foundation  
Chairman of the Board of Directors, MBI International

**MICHIGAN STATE**  
**UNIVERSITY**

June 24, 2009

D. Bernie Steele, Ph.D.  
Director-Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

RE: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096:  
Demonstration of Integrated Biorefinery Operations

Dear Dr. Steele:

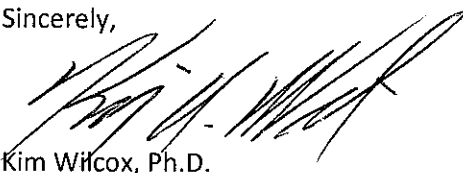
This letter is to confirm Michigan State University's commitment to participate in the world class team headed by MBI International to demonstrate a pilot-scale AFEX Integrated Biorefinery in response to the DOE solicitation referenced above.

As a partner in this project, MSU faculty and professional staff will assist with feedstock logistics, facility and process design, life cycle analysis, modeling and updating of the Business and Commercialization Plan. MSU personnel will also participate in quarterly meetings and StageGate reviews.

Using the University's normal accounting practices, we have determined the budget for our portion of the project to be \$422,676 which is approximately 1.8% of the total project budget. MSU expects to receive \$244,245 in project funding, and will contribute \$178,431 (approximately 0.75% of the total project cost) as cost share in the form of in-kind services.

We look forward to working with you and the excellent team that MBI has assembled to demonstrate the AFEX Integrated Biorefinery at pilot scale and to ready it for commercialization.

Sincerely,



Kim Wilcox, Ph.D.  
Provost and Vice President for Academic Affairs  
Michigan State University



OFFICE OF THE  
**PROVOST**

Michigan State University  
Administration Building  
East Lansing, Michigan  
48824-1046

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1210 Vermeer Road East  
Plant 1 P.O. Box 200  
Pella, IA 50219  
Phone: (641) 628-3141  
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June 17, 2009

D. Bernie Steele, Ph.D.  
Director-Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910

RE: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096:  
Demonstration of Integrated Biorefinery Operations

Dear Dr. Steele,

This letter is to confirm Vermeer's commitment to participate in the world class team headed by MBI International to develop a pilot-scale AFEX Integrated Biorefinery in response to the DOE solicitation referenced above.

As a partner in this project, Vermeer will provide the processed corn stover necessary for this project. We will procure, process to specifications and deliver the corn stover to the processing plant in Lansing, Michigan over the term of the project. We are familiar with your delivery requirements of 3 tons in the first year, 12 tons in the second year, 25 tons in the third year and 20 tons in the fourth year. Vermeer personnel will also participate in quarterly meetings and StageGate reviews during the project.

Using our usual cost structure, we have estimated our total budget at \$68,700, of which \$35,750 will be requested in project funds and \$32,950 will be contributed as cost share in the form of in-kind services. We understand that this cost share contribution is equal to approximately 0.14% of the total project budget of \$23,487,796.

We look forward to working with you and the excellent team that MBI has assembled to demonstrate the AFEX Integrated Biorefinery at pilot scale and to ready it for commercialization. We are particularly interested in the concept of Regional Biomass Processing Centers described in the proposal. We understand that such Centers could treat a variety of feedstocks in local communities and deliver densified pretreated feedstocks to a central bioconversion facility. We believe that such a concept would address the supply chain issues inherent in many plans for cellulosic fuel production.

Sincerely,

  
SR Proj Engineer  
Vermeer Corporation  
Pella, Iowa



**The Andersons, Inc.**

P.O. Box 119 • Maumee, Ohio 43537 • 419/893/5050

February 9, 2009

Bobby Bringi, Ph.D.  
President and CEO  
MBI International  
3900 Collins Road  
Lansing, MI 48910

Dear Dr. Bringi,

We are providing this letter to you, at your request, in support of US Department of Energy Grant DE-FOA-0000096 entitled Demonstration of Integrated Biorefinery Operations.

The Andersons, Inc. is a publicly traded company with diverse interests that include agribusinesses such as grain and plant nutrients as well as railcar leasing and repair, industrial products formulation, turf products, retailing and most recently, ethanol operations. The Grain Division operates grain terminals in Ohio, Michigan, Indiana, and Illinois with storage capacity of nearly 90 million bushels. The division trades and merchandises more than 250 million bushels of grain in the Eastern Corn Belt and Canada. The Ethanol Division operates three plants; a 55 million gallon plant in Albion, Michigan, a 110 million gallon plant in Clymers, Indiana and a 110 million gallon plant in Greenville, Ohio.

We are interested in improving the economic potential and diversity of our emerging ethanol operations. We continue to monitor progress in the field of cellulosic ethanol, and if the market and technology factors make it economically viable in the future, subject to our management's approval, we would be interested in evaluating investments in cellulosic ethanol technologies, especially to the extent that they synergize with our current assets.

We are familiar with the proposal being prepared by MBI International and its technology partners, Michigan State University (MSU) and the National Renewable Energy Laboratories (NREL) of Golden, Colorado. We have followed the progress at MBI with respect to the AFEX pretreatment technology, which is being developed in collaboration with Dr. Bruce Dale at MSU. We are familiar with the proposed Plan for Commercialization that incorporates the concept of Regional Biomass Processing Centers. We understand that these Centers could pretreat a variety of feedstocks in local communities and deliver densified pretreated feedstocks to a central biorefinery for fermentation and ethanol production as well as offer pretreated feedstock for sale to animal owners for energy feeds.

We believe that the concept of Regional Biomass Processing Centers has merit, and is worthy of the "de-risking" efforts that your team is proposing. We believe that your commercialization plan could (i) address several supply chain challenges inherent in many plans for cellulosic fuel production and (ii) provide rural development benefits for communities growing energy crops or harvesting crop residues.

We concur that a pilot plant, operating in a continuous manner and recycling the ammonia catalyst, is an important element to de-risk the AFEX pretreatment process so that process costs and process efficiencies can be incorporated in a model for an integrated biorefinery.



**The Andersons, Inc.**

P.O. Box 119 • Maumee, Ohio 43537 • 419/893/5050

It is our understanding that this project proposal is designed to accomplish this objective and provide the basis for moving toward a Demonstration Plant.

To the extent that the proposed project develops the information necessary for a commercial opportunity in cellulosic ethanol production, we may be interested in participating in a demonstration plant for cellulosic ethanol production as an addition to one of our existing facilities.

We wish you success on your proposed project, and look forward to being connected and monitoring progress in this area of vital regional, national and global importance.

Sincerely,

The Andersons, Inc.

By: 

Its: VP GM Ethanol U.S.

June 17,2009

D. Bernie Steele, Ph.D.  
Director-Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

RE: U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096: Demonstration of Integrated Biorefinery Operations

Dear Dr. Steele:

This letter is to confirm Novozymes commitment to participate in the world class team headed by MBI International to develop an AFEX Integrated Biorefinery at pilot scale in response to the DOE solicitation referenced above.

As a partner in this project, Novozymes will perform limited research and development to select enzymes that are most efficient in converting AFEX-treated corn stover to monomeric sugars. Novozymes personnel will be involved in techno-economic modeling, life cycle analysis, and shakedown and capstone runs to demonstrate the proposed technology. They will also participate in quarterly meetings and StageGate reviews. Novozymes will also provide all enzymes required for the project.

Using our normal cost structure, we expect our contribution to this project to equal \$143,437. The full amount will be contributed as cost share in the form of in-kind services. This contribution will come from employee's time, overhead charges, travel and enzyme costs. We understand this contribution is equal to 0.61% of the total project budget of \$23,487,796.

We look forward to working with you and the excellent team that MBI has assembled to demonstrate the AFEX Integrated Biorefinery at pilot scale and to ready it for commercialization.

Sincerely,

By: 

Title: VP of R&D

Novozymes North America, Inc.  
77 Perry Chapel Church Road  
P.O. Box 576  
Franklinton, North Carolina 27525



June 20, 2009

D. Bernie Steele, Ph.D.  
Director-Operations  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

Dear Dr. Steele:


ICM is the nation's leading provider of ethanol process design and engineering, with 101 dry-mill fuel ethanol plants operating in the United States and Canada that use ICM ethanol process technology. ICM-designed plants consistently perform 24 hours per day, at least 353 days per year for a 96% runtime – the highest performance measure in the industry. ICM's market success, over 50% of market share in current ethanol production, results from the fact that ICM process technology simply runs better, faster, cheaper and with less downtime than competing technologies. ICM employs 30 full-time engineers in its various departments, including process chemical engineers, mechanical engineers, electrical engineers, and civil engineers. ICM also designs, engineers, and installs a proprietary distributive control system (DCS) for ethanol plants that use ICM process technology. ICM employs another 30 scientists and operators in its two R&D laboratories and pilot plant.

We are aware that MBI and its technical partners Michigan State University, Novozymes Inc, Vermeer Corporation, and The National Renewable Energy Laboratory are preparing a submission for U.S. Department of Energy Funding Opportunity Announcement DE-FOA-0000096. We understand this submission is for a grant to build and operate an AFEX Integrated Biorefinery pilot plant making ethanol from corn stover at 1 ton per day. This biorefinery will utilize the AFEX treatment process, an innovative treatment process that uses ammonia as a catalyst.

Our scientific and engineering experts have reviewed the Process Flow Diagrams and the design engineering work for the proposed AFEX treatment pilot plant that will be a part of the proposal. We believe this plan, in the design engineering phase, is an acceptable plan for a continuous operating system and an ammonia recovery system. We look forward to providing input as these plans move towards construction, and we have an interest in providing engineering services if an award is granted by the DOE.

We also understand that one of your proposed marketing plans could be to partner with other technology providers to market the AFEX integrated process as part of a technology package offered to interested commercial parties. ICM would be most interested in such a marketing venture, as technology integration and successful delivery of technology to market is the main direction for ICM. We have been major technology providers to the dry mill ethanol industry and we plan to have the same impact in the developing the cellulosic ethanol industry. We look forward to working with MBI to this end.

Sincerely,



Douglas B. Rivers, Ph.D.  
Director, Research & Development

**Chris Fomey**  
Airgas Specialty Products  
Account Manager  
7452 Claremont Street  
Canton, MI 48167  
CELL (734) 276-7877 eFax (877)349-0207  
<http://www.airgas.com>  
[chris.fomey@airgas.com](mailto:chris.fomey@airgas.com)

June 22, 2009

Bobby Bringi, President & CEO  
MBI International  
3900 Collins Road  
Lansing, MI 48910-8596

Dear Dr. Bringi:

Airgas, Inc. (NYSE: ARG), Radnor, PA, through its subsidiaries, is the largest U.S. distributor of industrial, medical, and specialty gases. Airgas, Inc. is also a leading U.S. distributor of safety products, the largest U.S. producer of nitrous oxide and dry ice, the largest liquid carbon dioxide producer in the Southeast, and a leading distributor of process chemicals, refrigerants and ammonia products.

Ammonia is supplied through Airgas Specialty Products (ASP) of Duluth, GA. ASP, formerly the Atlanta-based Industrial Products Division of LaRoche Industries, is a leading distributor of anhydrous ammonia and related services in the United States. ASP's network of 24 ammonia distribution, field service and office locations, focuses exclusively on industrial applications and serves over 3,000 customers. The company is a primary supplier of ammonia to the fast-growing market segment of power plants, oil refineries and other manufacturing facilities nationwide that require ammonia for nitrogen oxide removal. ASP also serves the metal treating, heat treating, chemical processing, industrial refrigeration, and water treatment industries. Ammonia is one of the most highly used inorganic chemicals in the world today. Worldwide ammonia production in 2004 was 109 million metric tons, according to the United States Geological Survey.

Airgas Specialty Products offers safety training through its Ammonia E-training Program, which is a mixture of on-site, off-site, and Internet-based training customized to your company's training requirements and resources. ASP will develop a program of documented training for each individual who must be trained.

Training is available in the following areas:

- Anhydrous Ammonia Awareness
- Aqua Ammonia Awareness
- Aqua Ammonia and Anhydrous Ammonia Handling-Details
- Ammonia Properties
- Ammonia Safety

These programs will meet OSHA PSM, EPA RMP, and other safety requirements.

#### RISK MANAGEMENT PLANS

Airgas Specialty Products will develop a PSM/RMP Program uniquely fitted to your company's existing programs and policies. A PSM/RMP program developed for your company will meet all OSHA PSM requirements, EPA RMP requirements, and California CalARP requirements.

Since ammonia is our "core" business, we can often provide on-site ammonia equipment maintenance faster and more cost-effectively than internal staff. You can be assured that our technicians follow all of the latest in PSM/RMP/OSHA safety guidelines.

Services Offered:

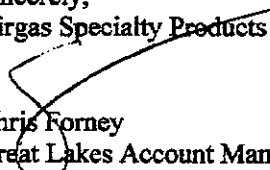
- Ammonia Quality Testing
- System Pumpouts
- Storage System Inspections/Repairs
- Tank Refurbishments/Refits
- Complete System Maintenance
- Leak Repair

We are aware that MBI and Michigan State University are preparing a submission for U.S. Department of Energy (DOE) Funding Opportunity Announcement DE-FOA-0000096. We understand this submission is for a \$18.8 million grant from the DOE to demonstrate fuel production from corn stover using the AFEX process, a novel pretreatment that uses anhydrous ammonia as a catalyst. We also understand this grant requires a 20% cost share from the participants.

Our engineering group has reviewed the Process Flow Diagram for the proposed AXEX pretreatment pilot plant that will be a part of a proposal. We find this plan, in the design engineering phase and subject to the comments made by our engineers, is an acceptable plan for an ammonia recovery system. We look forward to providing input as these plans move towards construction and will give advice on the safety and regulatory obligations necessary to provide safety to workers and meeting regulatory standards. We also have expertise in recovery of contaminated ammonia and can offer services and advice on how to deal with potential contaminants in the recycled ammonia as it is recovered and reused.

Airgas Specialty Products supports its customers with the above services and advice and we are happy to offer these services to you as you proceed with this project.

Sincerely,  
Airgas Specialty Products

  
Chris Forney  
Great Lakes Account Manager  
Airgas Specialty Products  
Canton, MI 48187  
[www.airgasspecialtyproducts.com](http://www.airgasspecialtyproducts.com)