**Developing a Biomass Industry in North Dakota**
**NDSU/MBI International Interim Report through December 2008**

## Background

The economic potential of bio-based products (materials, fuels, chemicals) is substantial. A consortium led by NDSU is currently engaged in a project to commercialize technology, developed by MBI International Lansing, MI, for producing cellulose nanofibers (very small fibers) from wheat straw. The cellulose nanofibers (CNF) would then be used to make bio-composites that could substitute for fiberglass and plastics in many applications, including automotive parts. The first major milestone in the commercialization effort is to address key engineering and economic questions to determine the technical and economic feasibility of a pilot scale production process while at the same time analyzing the integration of components made from biomaterials into the supply chain. These analyses are prerequisite to the construction and operation of a pilot plant to demonstrate the commercial potential of this technology.

The intent of the work undertaken with Industrial Commission funding is to extend the NDSU/MBI project by (1) completing the detailed investigations necessary to define value propositions needed to move the project from the investigation stage to the development stage and (2) further refining the strategic plan for industry development.

## Objectives

The goal of this project is to develop a strategic plan for establishing a biomaterials industry in North Dakota. Initial efforts will be focused on technical (Objective 1) and economic (Objective 2) requirements for commercializing technology to produce bio-based cellulose nanowhiskers. MBI is focused on the following objective:

1. Completing the detailed investigations necessary to define (a) a scalable process design, (b) mass and energy balances necessary to determine the cost of the process, (c) a procedure for qualitative and quantitative analysis of the structural materials available from wheat straw, and (d) a system for analyzing the structural enhancements of polymers from the inclusion of wheat straw fibers.

The NDSU study team is focusing on the second objective:

2. Refining the initial investment analysis for the business as data is added to key parameters regarding capital costs and manufacturing yields and preparing 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 for a corporate entity as well as examine potential markets, capitalization requirements, and projected financial performance.

## Approach

### Objective 1

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 will address 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
   - AFEX processing conditions including temperature, pressure, ammonia loading and retention times
   - Enzyme hydrolysis conditions including screening of enzymes for more efficient conversion
   - Screening of organisms capable of utilizing both 5 and 6 carbon sugars

2. Determine the best extraction methods for refining cellulose nanofibers from wheat straw fermentation residues
   - Define the process
   - Determine the mass balances
3. Detailed investigation of the production of biocomposites from wheat straw nanofibers. The program will investigate:
   - Potential uses of enhanced polyvinyl alcohol (PVOH) films
   - Expansion of resin use beyond PVOH
   - Production of test bars for comparative analysis
   - The production of waste streams and potential use/disposal of such streams
   - Determination of mass balances for the process
4. Economic modeling
   - Update the current models with mass balances and current economic data
   - Develop a preliminary pilot process design

## Highlights of major activities in the reporting period:

The CAFEX II pretreatment system has been designed and constructed (Figure 1). All safety systems are installed and tested. Initial trials have been conducted to assess operational processes including: 1) feeding and pumping of biomass; 2) introduction of moisture and ammonia; 3) mixing of biomass with water and ammonia; 4) mixing and heating; and 5) maintenance of temperature and pressure within the reactor. In this system ammonia is not recovered. Samples are taken at the end of the reactor and the rest of the material is quenched with water and acid and discarded.

Our approach has been to demonstrate the CAFEX concept using DDGs and corn Fiber, which can be pumped using current equipment, while at the same time developing techniques to pump high-impact feedstocks. Tests with DDGs in the continuous reactor during the reporting period revealed problems with pressure and temperature control. With the original configuration, we typically observe a relatively stable temperature that is significantly lower than the target, and widely fluctuating reactor pressure. Possible causes for the poor temperature and pressure control include: 1) ammonia flashing in the low pressure region of the annular jet pump, 2) poor mixing of ammonia with moist biomass, causing flashing of ammonia-rich zones at the point of steam injection in the annular jet pump, 3) interaction of the flow pulses from the lobe pump at the discharge end of the reactor with flow pulses from the progressive cavity pump at the feed end of the reactor, causing pressure oscillations and flashing within the incompressible biomass/ammonia/water mixture in the reactor. Several experiments were run to isolate and, when possible, eliminate each of the three possible causes mentioned above.

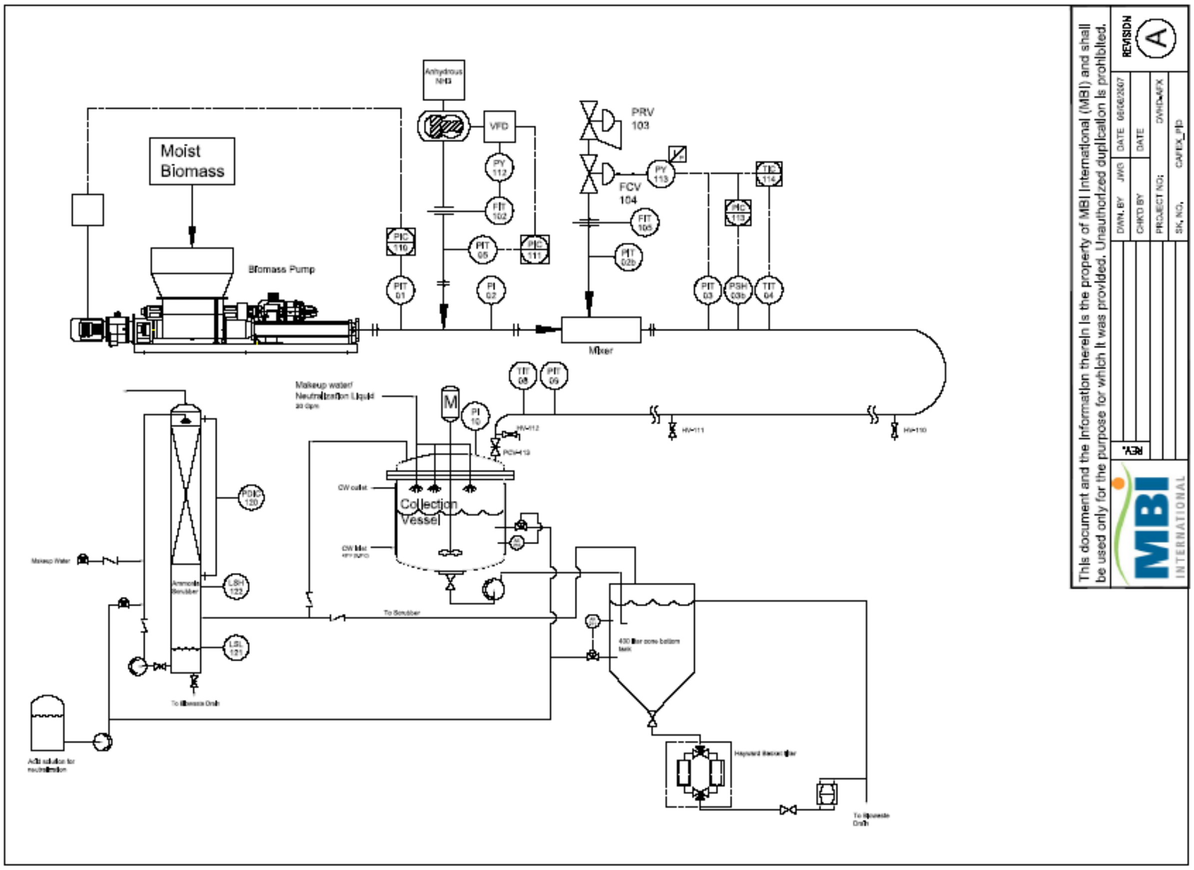


**Figure 1. Schematic of CAFEX II Pretreatment System**

In this period, the physical characteristics of wheat straw such as water absorption saturation capacity and hydraulic conductivity were measured and the results are summarized in Table 1.

**Table1. Water absorption saturation capacity and hydraulic conductivity of wheat straw**

| 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 |

Pumpability of wheat straw slurry (particle size of 1-2 mm) was tested with our current pump (Seepex BTI 5-24). Even at high moisture levels the current pump was not able to pump the wheat straw slurry. However, using additives to modify the slurry properties improved the pumpability of the wheat straw. Currently we are testing different additives at different levels to increase the flowability of wheat straw.

A different model of pump (Seepex BTH 17-12) has been ordered and the unit will be tested with wheat straw by end of January 2009. For this test a ton of wheat straw has been ordered from Ohio and arrangements have been made with Prater-Stering to have the wheat straw ground to 2mm particle size.

Quantitative recovery of $NH_3$ from AFEX-pretreated wheat straw was investigated during this period. Wheat straw, water, and $NH_3$ were charged into 1-gallon Parr vessel. AFEX temperature and pressure was maintained for 15 – 30 minutes. Vessel pressure was released, and $NH_3$ flashed into a Citric Acid trap (Trap 1). Purge gas ($N_2$ or air) was flowed through the vessel into another Citric Acid trap (Trap 2). If necessary, purge gas was continued into a separate Citric Acid trap (Trap 3). Citric Acid was back-titrated to quantify mass of $NH_3$ trapped. The results are presented in Table 2 and Figure 2.

**Table 2. Recovery of ammonia from AFEX-pretreated wheat straw**

| Run# | WS0 | WS1 | WS2 | WS3 | WS4 | WS5 | WS6 |
|---|---|---|---|---|---|---|---|
| Date | 2-Jun | 3-Jun | 4-Jun | 5-Jun | 9-Jun | 11-Jun | 13-Jun |
| Wheat straw charge (g) | 0 | 150 | 150 | 150 | 150 | 150 | 150 |
| Water charge (g) | 0 | 150 | 151 | 150 | 150 | 550 | 64 |
| Ammonia charge (g) | 143 | 125 | 142 | 144 | 145 | 160 | 107 |

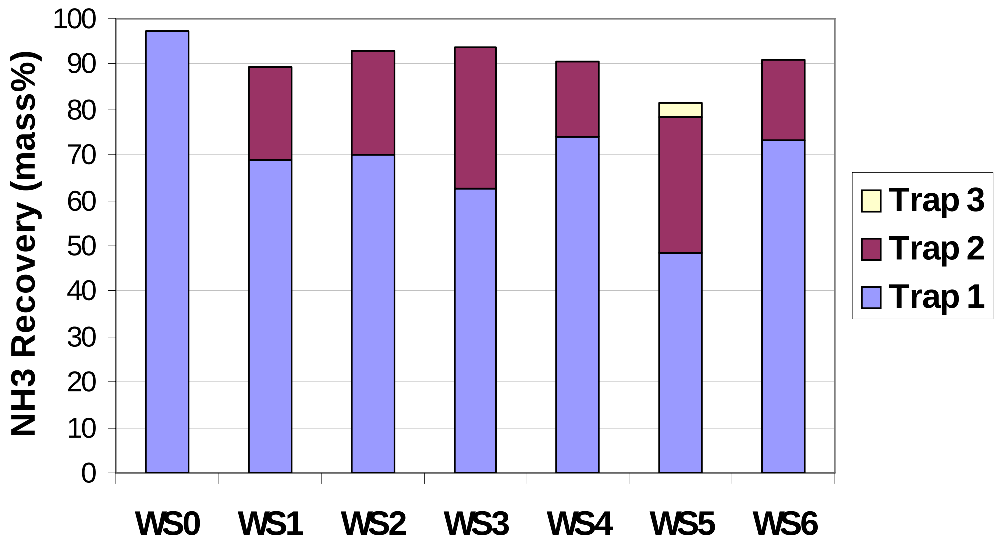


**Figure 2. Recovery of ammonia from AFEX-pretreated wheat straw**

MBI has executed the subaward to Michigan State University for development of biocomposites from wheat straw.

**Plan to Proceed**

MBI will be testing the new pumping system in January of 2009. Once adequate pumping is established we will proceed with the validation of each unit operation of the CAFEX II system. Once operational we will begin producing material for Michigan State University to develop biocomposite materials.

During December of 2008, the Department of Energy released a request for proposal to develop a fully integrated pilot plant to prove the feasibility of promising new technologies for conversion of lignocellulosic materials to biofuels. Several projects are expected to be funded and individual awards can be as much as $16 M plus 30% cost share. MBI plans to compete for this funding. The planned facility would fully integrate biomass processing and production of biofuels. This would allow us to fully integrate and test the ammonia recovery aspect of the AFEX process.

Objective 2

The information developed in steps 1- 4 (above) will be used to develop a strategic business plan that details the likely nature of operations and corporate organization for a commercial entity as well as examine potential markets, capitalization requirements, and projected financial performance. This business plan will be prepared so as to be readily usable by potential partners for investment analysis to facilitate the commercialization process. The study team will identify potential private sector investors who have a strategic interest in commercializing this technology (e.g., potential users of material, energy companies, engineering firms). Based on interaction with potential investors, the plan will be refined, with the goal of achieving commitment by investors and stakeholders that will provide for the long-term implementation of the plan, construction of a commercial biorefinery in North Dakota.

Highlights of Activities during Reporting Period

Activities have focused on identifying potential private sector investors who have a strategic interest in commercializing this technology (e.g., potential users of material, energy companies, engineering firms). Based on interaction with potential investors, the plan will be refined, with the goal of achieving commitment by investors and stakeholders that will provide for construction of a commercial biorefinery in North Dakota. Initial interactions have been encouraging, and more detailed discussions with one or more investors will be pursued in the coming months. The study team has also begun to investigate public sector resources that could be mobilized for a commercialization effort. Federal programs included in the EISA of 2007 and in the 2008 Farm Bill appear to hold promise, although in some cases program details have not been announced.

Plan to Proceed

The study team will complete the investment analysis and strategic business plan.