# Quarterly Update



National Bioenergy Center Biochemical Platform Integration Project

Biomass Program—Sustainable Fuels, Chemicals, Materials, and Power

July-September 2009, #24

The Biochemical Process Integration Task focuses on integrating the processing steps involved in enzyme-based lignocellulose conversion technology. This project supports the U.S. Department of Energy's efforts to foster development, demonstration, and deployment of "biochemical platform" biorefineries that economically produce commodity sugars and fuel ethanol, as well as a variety of other fuel and chemical products, from abundant renewable lignocellulosic biomass.

The National Renewable Energy Laboratory manages this project for DOE's Office of the Biomass Program. Information on the Biomass Program is available at Biomass Program.

To discuss the contents of this update, or for further information on the Biochemical Process Integration Task, contact Dan Schell at NREL, phone (303) 384-6869, email dan.schell@nrel.gov

## R&D Progress Biomass

#### **Automating Biomass Compositional Analysis**

Automating wet chemical compositional analysis of lignocellulosic biomass materials will significantly improve sample throughput and perhaps measurement accuracy. Being able to rapidly measure the composition of many samples will expedite the development of NIR-based models to predict biomass composition. Using these models for real-time measurements will enhance process and quality control and thus enable higher conversion yields. In addition, higher sample throughput will improve research productivity at NREL and other institutions.

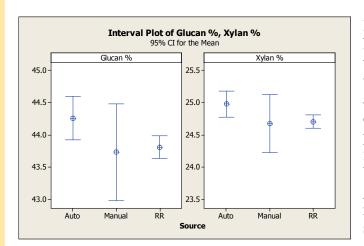


Figure 1. Glucan and xylan measurement of a single corn stover sample using the robot (Auto) versus the traditional wet chemical analysis method (<a href="http://www.nrel.gov/biomass/pdfs/42618.pdf">http://www.nrel.gov/biomass/pdfs/42618.pdf</a>) performed either by a single analyst (Manual) and or a team of analysts (RR).

In the summer of 2008, NREL acquired a robotic workstation and began work to automate the analysis procedure for determination of polymeric carbohydrates in lignocellulosic biomass. Figure 1 shows glucan (cellulose) and xylan content of a corn stover sample measured manually (i.e., using traditional laboratory equipment) or using the robotic workstation. In each case, the analysis was performed using the standard two-stage acid digestion procedure

shown in Figure 2. The steps in the dashed box in Figure 2 are the ones currently being automated. During this work, we optimized performance of the solid and liquid dispensing systems, operation of the digestion reactor, and filtration of acid-insoluble lignin. Statistical tests demonstrated that the accuracy of the measured polymeric carbohydrates using the robot was equivalent to the manual analysis method. Future work will further develop the ability to perform the acid insoluble lignin measurement.

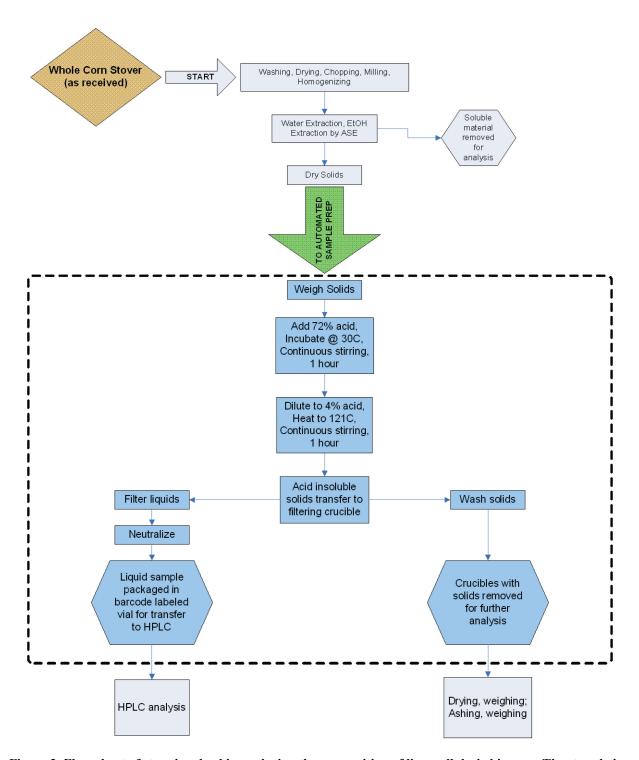


Figure 2. Flow chart of steps involved in analyzing the composition of lignocellulosic biomass. The steps being automated, i.e., performed by the robot, are shown within the dashed lines.



### **Analyzing Acid-Soluble Lignin in Biomass Hydrolysates**

We are continuing efforts to improve the measurement of acid-soluble lignin (ASL) in pretreatment biomass hydrolysate liquors. Previous work demonstrated that sugar degradation products interfere with the standard UV spectroscopic method for measuring ASL (http://www.nrel.gov/biomass/pdfs/42617.pdf). The inability to accurately measure ASL is partly responsible for high lignin mass balance closure across dilute-acid pretreatment. Three new UV-Vis methods were investigated to determine if they would produce a better ASL measurement, but none of these methods proved to be satisfactory. We also investigated a liquid-state Nuclear Magnetic Resonance (NMR) method. However, it was difficult to estimate ASL content by <sup>1</sup>H-NMR because integration of the low NMR signal assigned to the aromatic proton region was hampered by baseline distortions and low ASL content. On the other hand, well-separated carbon signals from lignin aromatic structure appeared in the quantitative <sup>13</sup>C-NMR spectrum of the hydrolysate liquor. Based on the peak area of the lignin aromatic carbon, the ASL content of a corn stover biomass hydrolysate was estimated to be 18.9% of original lignin content, which was less than the ASL content measured by the spectroscopic method (26.3%). This result demonstrated that an NMR method for determining ASL content could be developed, but the <sup>13</sup>C-NMR technique requires long NMR scan times. However, <sup>13</sup>C-NMR may be useful in developing correlation curves between UV data and NMR data to allow more accurate measurements of ASL using UV spectroscopy.

#### **Biochemical Process Integration Task Information**

Web-based information on the process integration project, including presentations made at past review meetings, are available at the following links: <a href="http://obpreview07.govtools.us/biochem/">http://obpreview07.govtools.us/biochem/</a> and <a href="http://www.obpreview2009.govtools.us/biochem">http://www.obpreview2009.govtools.us/biochem</a>). A discussion of how Stage Gate management is used in the Biomass Program is also available at this site (Stage Gate Management).

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