Quarterly Update

National Bioenergy Center Sugar Platform Integration Project

Biomass

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

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The Sugar Processing 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 "sugar platform" biorefineries that produce inexpensive 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 information in this update or for further information on the Sugar Processing Integration Task, contact Dan Schell at NREL, phone (303) 384-6869, email dan_schell@nrel.gov

Two Year Anniversary.

This volume marks the end of our second year of publishing this newsletter. I would like to thank all of our readers and in particular those that have provided comments over the years. We will continue our efforts to provide technical updates on our research progress as well as highlights from other related and interesting work at NREL.

28th Symposium on Biotechnology for Fuels and Chemicals.

Mark your calendars as the 28th Symposium is rapidly approaching with abstracts due in early December. The Symposium will be held in Nashville, TN on April 30-May 3, 2006.

R&D Progress Biomass

New Analytical Method for Biomass Moisture Measurement.

One objective of our work is to develop new analytical methods for analyzing biomass samples that improve accuracy and reduce labor requirements when compared to traditional measurement techniques. Recently, we investigated Karl Fischer (KF) titration as a technique to selectively measure water in pretreated biomass slurries and liquors. Moisture measurements via oven drying methods are accurate for raw feedstocks, but are problematic when applied to pretreated materials due to loss of volatile compounds, other than water, during the drying process. A further benefit is a reduction in analysis time from 24 hours for oven drying, or 30 minutes to one hour for infrared moisture balances, to 3 to 5 minutes for KF titration. Preliminary results suggest that KF titration is more accurate than traditional moisture measurement techniques for pretreated materials, but validation work is required before this method will be deployed as a Laboratory Analytical Procedure (LAP).

New Work Advances Methods for Biomass Sugar Analysis.

Accurate and precise compositional analysis of biomass is critical to understanding and assessing biomass conversion technology. A high degree of confidence in both the wet chemical and rapid instrumental methods of analysis is required to enable accurate yield and mass balance calculations, which underlie sound cost estimates. In an effort to improve analysis methods, we evaluated several instrumental and/or detection schemes for measuring biomass-derived sugars using High Performance Liquid Chromatography (HPLC). Options evaluated included Evaporative Light Scattering Detection, Pulsed Amperometric Detection, new HPLC columns, and different mobile phase compositions. We determined that Shodex Sugar SP-0810 columns achieve enhanced baseline resolution and improve measurement accuracy when compared with older methods. High Performance Anion Exchange Chromatography with Pulsed Amperometric Detection also gave excellent

baseline separation for most biomass-derived sugars. We will validate and document these new techniques and update our LAPs.

Methods Identified for Potentially Improving Biomass Lignin Analysis. Previous task work demonstrated an inability to accurately close lignin mass balances during dilute acid pretreatment of corn stover. Therefore, we have begun to explore options for improving lignin measurements in materials derived from herbaceous feedstocks. Analytical methods in the current NREL biomass analysis portfolios for determination of lignin have been optimized for lignin in woody biomass, but have proven to be less reliable for accurately tracking lignin fractionation when processing feedstocks such as corn stover or switchgrass, largely due to the presence of protein and silica in these materials. Drawing from a depth of expertise in lignin chemistry within the National Bioenergy Center, and from the literature, several options were selected that may deserve further investigation. One option is to adapt analytical methods widely used in the food and feed industries, which use more dilute H_2SO_4 for digestion or replace H_2SO_4 with HCl to minimize condensation reactions between lignin and protein. Other solutions identified include combining the above digestion techniques with spectroscopic methods such as Fourier Transform Infrared, Near Infrared, Nuclear Magnetic Resonance and Pyrolysis/Mass spectroscopy to separately quantify lignin and other compounds such as silica and protein. Although some of the spectroscopic methods may be more cumbersome, expensive, and slow for routine analysis, all of them could be used to calibrate improved rapid biomass analysis methods for lignin and other herbaceous material components.

Initial Assessment Completed on Impact of Recycle Water on Process Performance. The main objective of this study was to assess the impact of water recycle on process performance. The goal was to recycle 25% of the stillage (bottom stream from ethanol distillation column) and have no negative impact on process performance. We used hydrolysate liquor from dilute sulfuric acid pretreated corn stover and fermented this material with *Zymomonas mobilis* 8b in batch fermentations and recycled stillage enough times (3) to approach steady state concentrations of non-sugar components. Performance was evaluated as a function of total solids concentration, that is, the effective total solids concentration of the pretreated material that was the source of the liquor, and fraction of stillage recycled (recycle ratio, RR). No impacts were observed at 15% or 20% total solids concentration with a RR of 10%. However, at 25% RR significant impacts were observed and ethanol yields at a 20% total solids and a RR of 25% were as low as 10%. Parallel studies with pure sugar solutions containing similar levels of acetic acid achieved good performance indicating that other inhibitors besides acetic acid are affecting performance.

Related Activities Biomass

Understanding the Rheology of High Solids Biomass Slurries. The rheological characteristics of liquids and slurries determine many of the flow and mass transfer properties of these fluids, and knowledge of rheological properties is useful to predict process performance and design equipment. Using plate-plate type rheological measurements under continuous shear, the rheology of pretreated corn stover (PCS) slurries was measured as a function of insoluble solids concentration and extent of pretreatment. Initial work has shown that the apparent viscosities of PCS slurries increase with increasing solid concentrations and that increased xylan removal decreases apparent viscosity. Thus, the availability of free water, which correlates with xylan removal, appears to play a significant role in determining the slurries' rheological characteristics. Ultimately, this effort

should substantially improve understanding of and cost estimates for high-solids biomass processing equipment. Future work will investigate slurry rheology during enzymatic cellulose hydrolysis and in collaboration with an engineering company recently placed on subcontract, we will generate design and cost information for high-solids processing equipment.

Sugar Processing Integration Task Information. Web-based information on the process integration project, including our presentations from the most recent stage gate interim review meeting, can be found at the following link (Process Integration Project Information). 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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