Rapid biomass analysis: New analytical methods supporting fuels and chemical production from biomass

Bonnie R. Hames*[a] Amie D. Sluiter[a] Chris J. Roth[a], and Robert R. Meglen[b]
[a] National Bioenergy Center, National Renewable Energy Laboratory
1617 Cole Blvd. Golden CO 80401
Fax: 303-384-6877, bonnie_hames@nrel.gov
[b] Latent Structures, 500 Manhattan Dr., B-8, Boulder, CO 80303

The ability to obtain an accurate chemical composition of biomass and biomass-derived samples using rapid and inexpensive methods is a key element supporting commercialization of processes that convert biomass to fuels and chemicals. The development and demonstrated use of rapid analysis methods for chemical characterization of feedstocks, process intermediates and solid residues will be presented. The new techniques being presented combine Near InfraRed (NIR) spectroscopy and Projection to Latent Structures (PLS) multivariate analysis for the rapid chemical characterization of hardwood, softwood and corn stover feedstocks. Methods will also be presented for the chemical characterization of biomass-derived solids produced in processes ranging from mild pretreatment to total saccharification.

Heterogeneity is an inherent characteristic of biomass feedstocks. Since variability of any biomass cannot be controlled, processes that use biomass feedstocks must be able to monitor the chemical composition of the feedstock and compensate for variability during processing. Chemical changes during the processing of biomass must also be monitored to ensure that the process maintains a steady state in spite of the feedstock variability. Finally, process residues and products must be evaluated to assess overall process economics. Current methods for chemical characterization of biomass feedstocks, process intermediates and residues are not applicable in a commercial setting because they are very expensive (labor intensive) and cannot provide analysis information in a time frame useful for process control [1]. For example, a complete analysis using traditional wet chemical methods cost $800-$1,000 per sample and results are not available for days. New techniques are needed to provide analytical support for large-scale processes that convert biomass to fuels and chemicals. One solution is to use established methods to calibrate rapid, inexpensive spectroscopic techniques, which can then be used for feedstock and process analysis. Near InfraRed (NIR) has several advantages over alternative spectroscopic tools since the samples require little, if any, preparation, the analysis can be done very rapidly, at very low-cost. Robust NIR instruments have been designed for process analysis, process control and field applications. Many of the NIR techniques that have been developed for use by the chemical, food processing and agriculture industries can be applied to the characterization of biomass. In contrast to the traditional methods, NIR/PLS techniques can perform the same analysis for about $10 per sample. A complete chemical characterization is available in a time frame relevant for process control, meaning that the information can be used to make the process adjustments necessary for steady-state production. The NIR/PLS methods described here provide these advantages without any loss of precision or accuracy relative to the calibration methods [2].

Process monitoring is one possible application of these rapid analytical techniques. **Figure 1** shows compositional changes during dilute acid pretreatment of poplar as measured by a NIR/PLS method calibrated for poplar process samples. Rapid analysis techniques allow processes operators to determine when the sample is depleted in xylan and is ready for the next process step, enzymatic hydrolysis. Stopping pretreatment early will leave xylan in the sample, which decreases enzyme efficiency. Continuing pretreatment beyond the point of xylan removal will result in loss of fermentable sugars, which could lower ethanol yields.

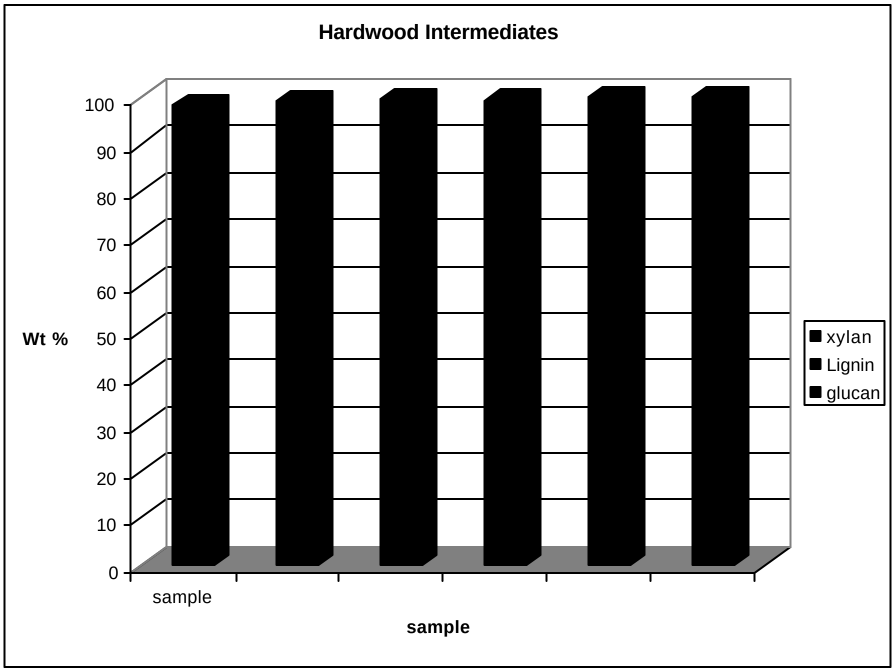

Figure 1. Compositional changes during dilute acid pretreatment of poplar as measured by a NIR/PLS method calibrated for poplar process samples

In addition to significant savings in time and money, rapid, inexpensive analysis techniques can be used to provide levels of information that were not previously available. For example, using a NIR/PLS technique calibrated for corn stover compositional analysis, approximately 200 samples from a bulk corn stover feedstock were analyzed in just a few days and at a cost of less than $2,000. This information was used to develop protocols for representative sampling of fields, bales, totes and bags of feedstock. The ability to accurately sample a bulk feedstock and to analyze hundreds of samples for about $10 each provides a new tool that is being used to assess the compositional variability of feedstocks as a function of variety, geographical location, storage and collection method. These studies would have been too costly to pursue without the savings in time and cost provided by NIR spectrometry.

Development of rapid NIR/PLS techniques includes, gathering appropriate calibration samples, chemical characterization of the calibration samples, developing spectroscopic methods for the rapid technique, PLS regression and validation of the PLS calibration model, developing QA/QC procedures and establishing guidelines for appropriate application of the new methods. Lessons learned in each of these areas will be presented.

Conclusions Rapid techniques for biomass analysis are a key element supporting commercialization of processes that convert biomass to fuels and chemicals. Rapid techniques based on NIR/PLS can provide significant savings in time and money with no loss of precision or accuracy relative to the calibration methods. These new rapid methods for biomass analysis can support and improve research by providing levels of information that would have been too costly to pursue using traditional wet chemical methods.

References [1] Milne, T.A., Brennan, A.H. and Glenn, B.H., Sourcebook of Methods of Analysis for Biomass and Biomass Conversion Processes, Eslevier Applied Science, New York, 1990
[2] DiFoggio, R., Examination of Some Misconceptions about Near-Infrared Analysis, Applied Spectroscopy, Vol. 49. Number 1, 1995, p. 67-75.