Biofuel / Biomass Feedstock & Conversion Research

Quality Assessment Research Unit
Russell Research Center
ARS-USDA, Athens, Georgia USA

Outline

- Accomplishments:
  - Cell wall structure of plants
  - Spectroscopic Analysis of feedstocks
    - Near-infrared of barley
    - Near-and mid-infrared of switchgrass
  - Enzymatic conversion (corn leaf, corn fiber and bermudagrass)
  - Analysis of Starch Structure with Gel-State NMR
  - Analysis of Down-Regulation of Lignin in Plants
- Planned Work:
  - Co-products from Flax
  - Microbial fermentation of C-6 & C-5 sugars
  - Spectroscopically assess additional perennial grasses
  - Identify new by-products from grasses
  - Assess structure/composition of lignocellulosic sources using spectroscopic imaging
- Instrumental capabilities
- Funding
- Cooperation

Cell Wall Structure
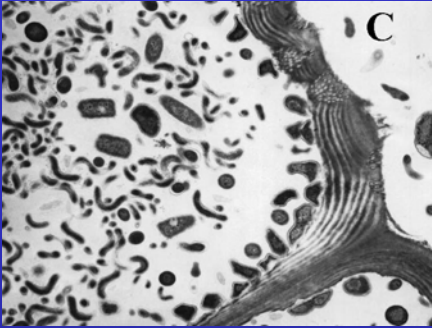
Rumen microbiology, plant structure, & improved forage use:
- new methods of cell wall analysis and assessment
- cell wall chemistry/structure related to recalcitrance
- methods to overcome recalcitrance, improve fiber nutritive value

Biomass research on agricultural sources re: bioenergy/ bioproducts:

- cell wall analysis and assessment

- cell wall chemistry/structure related to recalcitrance in bioenergy

- identification of lignocellulosic co-products related to bioenergy sources

- evaluation of consistency of materials using spectroscopy and chemometrics

Evaluation Products Using NIR (Prediction Error for Barley Analysis on Multiple Instruments)

Components		FT-NIR	XDS	NIR6500
Moisture		0.52	0.46	0.42
Starch		1.85	1.77	1.54
β-glucan		0.48	0.61	0.57
Protein		0.48	0.49	0.49
Oil		0.20	0.20	0.14
ash		0.14	0.13	0.12

Evaluation of Switchgrass NIR Chemometric Models (Using Partial Least Squares)

Dispersive NIR
(1100-2598 nm &10 nm Res.)

Components	# of outliers	LV	R^2	RMSECV
IVDMD	4	5	0.962	**2.50**
NDF	5	6	0.949	1.62
ADF	6	5	0.972	**1.28**
ADL	6	5	0.894	0.57
Total-N	6	4	0.985	**0.09**
Fat	8	7	0.912	0.20
Ash	5	7	0.934	**0.54**
Total-C	10	7	0.939	0.22

Fourier-transform NIR
(10,000-4,000 cm^{-1} & 4cm^{-1} Res.)

Components	# of outliers	LV	R^2	RMSECV
IVDMD	8	5	0.981	1.99
NDF	4	6	0.968	**1.78**
ADF	8	6	0.983	1.24
ADL	4	4	0.885	**0.64**
Total-N	11	4	0.991	0.08
Fat	4	8	0.971	**0.25**
Ash	8	6	0.960	0.50
Total-C	5	6	0.943	**0.26**

Best results are in red. Samples provided by Ken Vogel & chemical and dispersive NIR data were supplied by Gautam Sarath-ARS, Lincoln, NE.

Assessment of Native Starch Structure in Grains Using NMR Spectroscopy

**Apparent Amylose (%) versus 1-4/1-6 Integration Ratio**
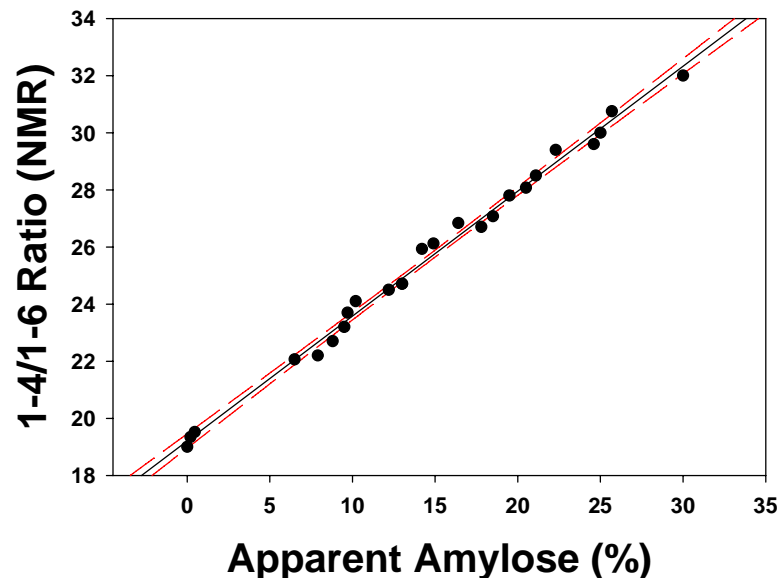
Range=0-30% Amylose

R^2=0.99

Y intercept=19.2 (Amylopectin branching ratio)

NMR calibrations have been developed for use as instrumental (primary) reference values for rapid analysis (NIR) of raw materials to enable "up-front" enzyme selection for starch digestion process.

Use of MIR and MS to Assess Down-regulation of COMT & CCoAOMT that Reduces Lignin
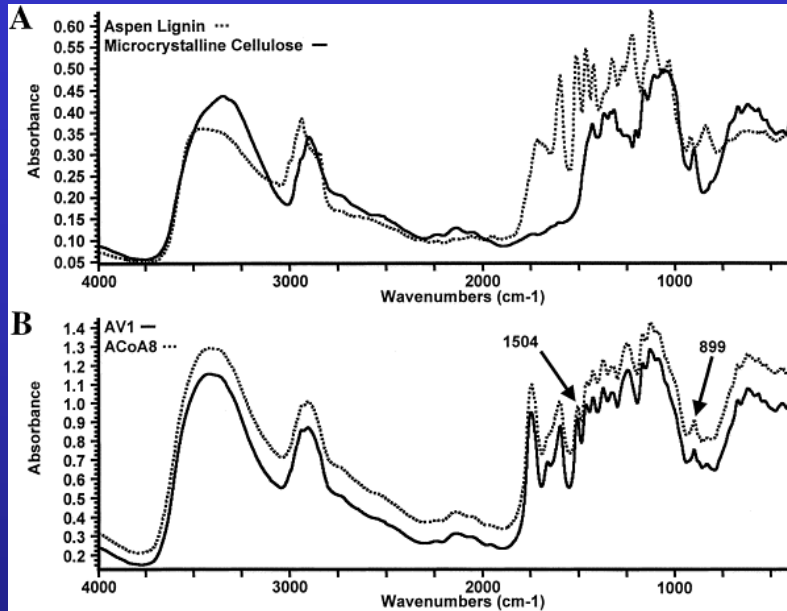
IR bands at 1508 and 899 cm-1 permitted the quantification of the effects of down-regulation at 56%.

In-source pyrolysis MS indicated the specific reduction of guaiacyl lignin units in transgenic tobacco plants cell walls
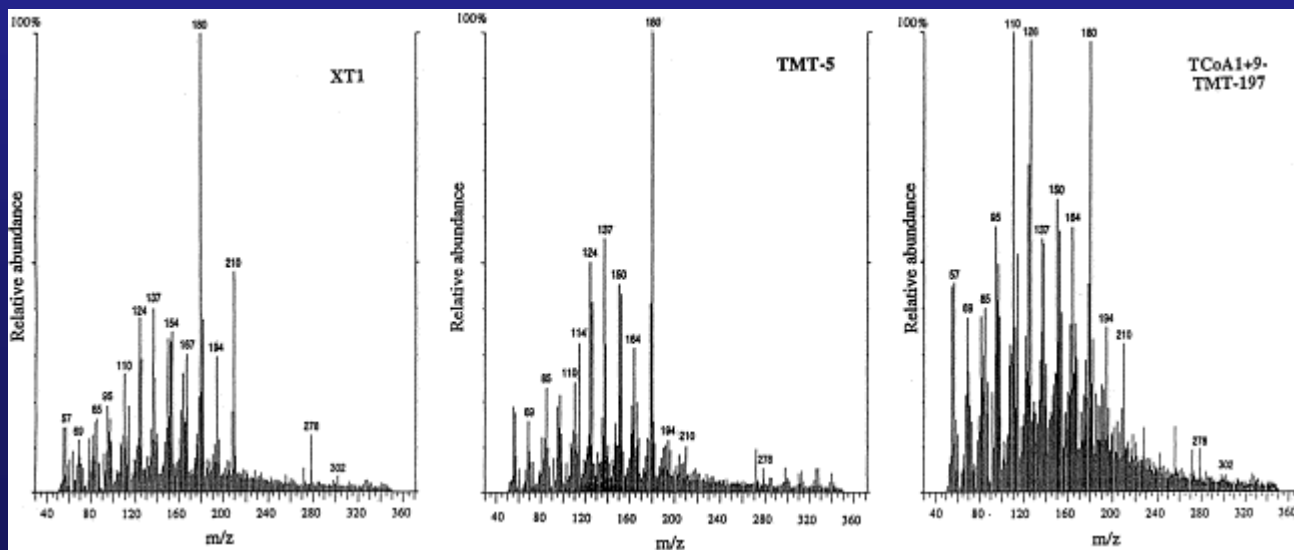
Industrial Opportunity for Bioenergy and Co-Products

Schweitzer Mauduit International (SMI)

- headquartered in Alpharetta, GA,

- the only flax fiber producer in NA today

- upgrade fiber for high-value uses

- increased shive as a waste product

- shive is cheap, already collected:

    bio-energy (thermal, syngas)

    extractable aromatic for antioxidants etc

    activated carbons (filtration)

ARS and SMI collaborations on retting, processing, standards work.

Opportunity for bioenergy and co-products.

Funding

- **Biomass ($275,000)**
- **Biofuels ($275,000)**
- **No separately coded projects only sub-coded (primary is NP306)**
- **3 GS-15 SYs Akin, Barton & Himmelsbach (@ 0.2-time each)**

Spectroscopic Instrumental Analysis Capabilities

Type	Analysis Function
Near-infrared (4)	Rapid Composition
Mid-Infrared (2)	Chemical Fingerprinting
Raman (1)	Chemical Fingerprinting
Pyrolysis-GC-MS (1)	Chemical Fingerprinting
Microscopes (UV, VIS, FL, IR & Raman)	Chemical & Structural
LC-NMR & solution, solid-state & imaging NMR	Chemical & Structural

Interactions / Collaborations

- Bill Anderson, ARS- Tifton GA; bermudagrass

- Ken Vogel/Gautam Sarath, ARS-Lincoln, NB; switchgrass

- Kevin Hicks, ERRL, Wyndmoor, PA; corn fiber/stover & barley

- Sevim Erhan, NCAUR, Peoria, IL; resins, chemical analysis

- Mike Cotta/Bruce Dien, NCAUR; enzymes, pretreatments

- Brian Condon, SRRC, New Orleans; enzymes, co-products

- Roy Dodd, Clemson Univ; products and co-products

- Maurice Snook, ARS-RRC; sugar/aromatic analyses

- Joy Peterson, Univ. GA; fermentation, microorganisms

- David Bransby, Auburn Univ; co-firing, thermal

- Enzyme companies: Novozymes, Genencor, Biocatalysts

- DOE Laboratories: NREL, Golden, CO; Idaho National Lab, Idaho Falls