

Prediction of Fermentable Starch Content by Near-Infrared Spectroscopy

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POET™

Introduction

- Rapid measurement of corn quality for ethanol production
- Fermentable starch measurement best indicates ethanol yield from corn
- Conventional fermentable starch measurement takes days
- Quick and easy measurement of fermentable starch content important to ethanol industry



Near-infrared Spectroscopy (NIRS)

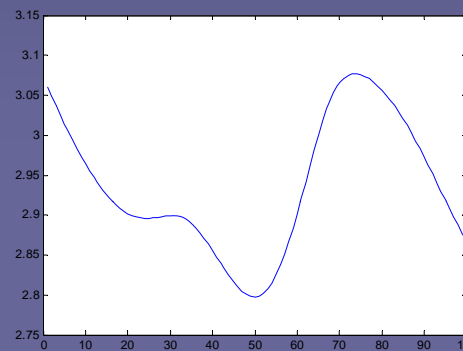
- Rapid and nondestructive technique
- Measures organic substances in seconds
- Common measurements for corn: protein, oil, starch, density
- Technology could be useful in rapidly identifying the value of corn for dry-grind ethanol production



NIRS Calibration

- Prediction model →

Spectra (100 pts)



$$\hat{Y} = b_0 + b_1x_1 + b_2x_2 + \dots + b_kx_k$$

Ethanol yield (gal/bu)

- Component model calculation →

Components

Protein (%)

Oil (%)

Starch (%)

Density (g/cc)

$$\hat{Y} = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4$$

Ethanol Yield (gal/bu)

Objectives

- Develop preliminary NIRS fermentable starch calibration using spectra and reference data from Illinois Crop Improvement Association
- Compare calibration to component calculation using multiple linear regression (MLR) analysis with combinations of protein, oil, starch, and density predicted from current Iowa State calibrations.



Reference Method

Fermentable starch measurement - Illinois Crop Improvement Association

- 249 corn samples
- 64 hour simultaneous saccharification and fermentation test
- Ethanol yield = loss in weight of sample from beginning to end of fermentation
 - $C_6H_{12}O_6 \longrightarrow 2C_2H_6O + 2CO_2 + \text{heat}$
- Presented in gallons/bushel of corn at 15% moisture
 - Mean=2.74 gal/bu
 - Standard Deviation=0.069
 - Range=2.55-2.89 gal/bu

<http://www.ilcrop.com/ipglab/corntest/corndesc.htm#EthanolFermen>



Methods



Calibration

- Partial Least Squares (PLS) spectra calibration model
- Infratec 1229 Grain Analyzer (FOSS Group, www.foss.dk), whole corn

Calculation

- Component calculation combinations (15) of protein, oil, starch, density
 - At 15% moisture
 - Predicted from Iowa State Infratec calibrations.
 - Could use any accurately calibrated NIRS unit.
- Models included 237 corn samples

■ Validation

- leave-one-out full cross validation
- external set of 55 samples representing a wide variety of samples

■ Software

- The Unscrambler 9.6 (CAMO Inc., www.camo.com)



Methods

- Statistics
 - Standard Error of Cross Validation (SECV): describes the precision, used in cross validation
 - Standard Error of Prediction (SEP): describes the precision, used in validation
 - R²: coefficient of determination, ranges from 0 to 1



Results

- Initial fermentable starch calibration and component calculation had similar results
- Calibration was slightly more precise, but the calculation may be more practical
- Combinations for calculation were not significantly different from each other
 - Protein, oil, density calculation in use
 - more reliable calibrations

Validation (new samples) Comparison

■ PLS fermentable starch calibration

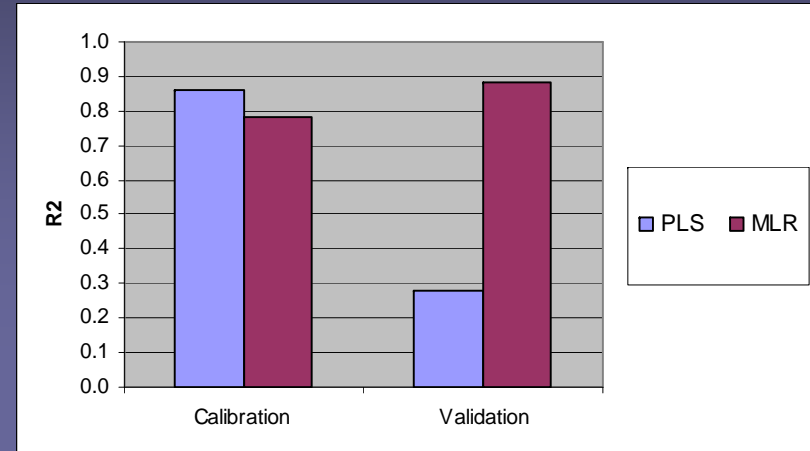
- poor at predicting ethanol yield
- Inconsistent with initial development

■ **SEP=0.40 (gal/bu)**
■ **R²=0.28**

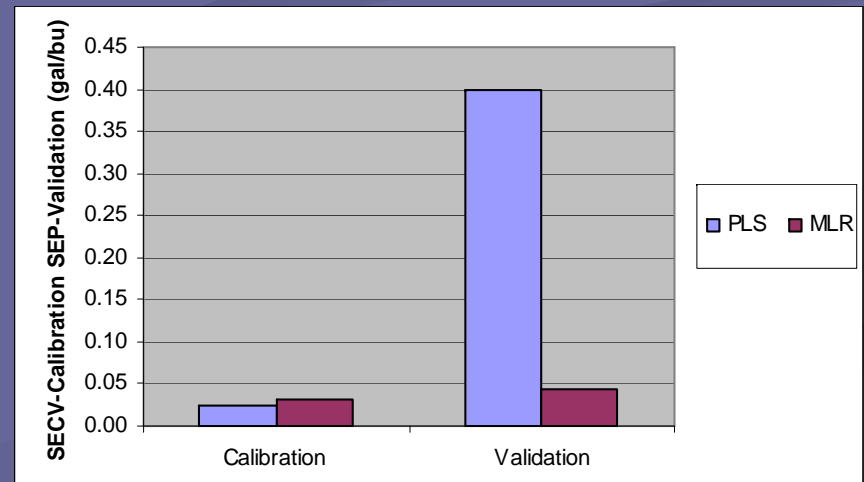
■ Component calculation

- consistent with initial development

■ **SEP=0.044 (gal/bu)**
■ **R²=0.88**



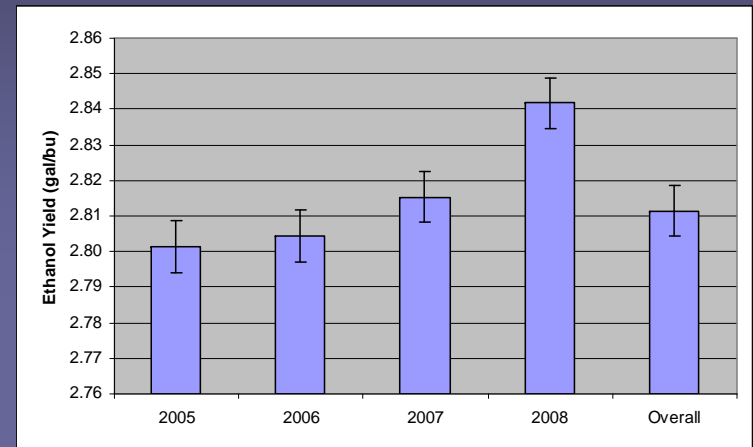
R² calibration and validation results for PLS and MLR



SECV and SEP calibration and validation

Calculation Applied to Plot Data

- Protein, oil, density calculation used to predict ethanol yield on past Iowa samples
- Increase in Ethanol Yield from 2005 to 2008
- 0.25 gal/bu range from year to year
- 2008
 - High ethanol yield
 - Low protein



Average Ethanol Yield for years 2005-2008

Year	n	Mean EY (gal/bu)	Stdev	Range
2005	436	2.80	0.05	0.27
2006	797	2.80	0.04	0.24
2007	536	2.82	0.04	0.21
2008	352	2.84	0.04	0.23
Overall	2121	2.81	0.04	0.29

Conclusions

- Component calculation performed better than the spectra calibration in validation (new samples).
- Implementing the component calculation based on current calibrations is easier than a new NIR calibration with better accuracy. Much less cost.
- Any NIRS unit can use constituent regression.
- Ranking for ethanol yield can be rapid, inexpensive, and accurate for all genetics.



Future Work

- Use equation for on-site trial at ethanol plant .
- Screen large number of samples for ethanol yield.
- Evaluate calculation's performance vs plant yield.



Acknowledgments

- Illinois Crop Improvement Association



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- Poet, LLC.

