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In-plant validation of an ethanol yield prediction equation

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In-plant validation of an ethanol yield prediction equation

by

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Gloria Starns
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ABSTRACT

Much of the fuel ethanol industry's current interest centers on maximizing ethanol yield and overall profits. This can be achieved by knowing the potential yield of input corn and working to identify what parameters are inhibiting reaching 100% fermentation efficiency. On average, ethanol plants produce 2.82 gallons of ethanol per bushel of corn, as compared to 2.51 gallons per bushel in 1994 (Renewable Fuels Association 2015). With the focus on improved starch production and access, corn quality is one of the best indicators of ethanol yield, as the amount of starch determines the theoretical amount of ethanol. Near-infrared spectroscopy (NIRS) is one such method that can be used to evaluate corn composition and, with an appropriate model, corn composition can be used to predict ethanol yield. Many current models are held back by real world applicability, in that they are restricted to lab-scale validation, direct NIRS calibrations, or proprietary models/equipment. At the commercial level, corporately-produced propriety models have been developed by DuPont Pioneer and Monsanto. Neither Monsanto nor DuPont Pioneer's products are available outside of company databases, and both are only applicable to Foss Infratec units, which left a need for a more universal method. Burgers et al. developed a multiple-linear regression equation for predicting corn ethanol yield based on near-infrared spectroscopy (NIRS) measurements of protein, oil, and density on a 15% moisture basis (Burgers, Hurburgh, and Jane 2009). Unlike corporately-developed models, this equation was intended to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used, as long as the calibration database was consistent. Iterations of the model were evaluated, and the most current version was chosen to use in the rest of the research. A comparison of the model predicted yield, based on inbound grain composition, and corresponding reported ethanol yield from the same grain was performed to validate the model.

The slopes for the plants' predicted and reported ethanol yields did not differ significantly from one another. Overall, the combined model for the linear regression produced a low R^2 value (0.23) which shows that a significant amount of variability in the data is not explained by the model. On average, the data validated the prediction model. Day to day or batch by batch variability in processing was not accounted for in the equation, but the variability of the corn composition was. From the linear regression analyses performed on each plant, the slopes are the same, but there is a plant-specific bias. This equation identified key corn quality parameters. Because the equation validated for all plants, the equation is validated to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used. The model validated with a root mean square error of 0.13 gal/bu, and no difference (0.0008 gal/bu) between overall reported and predicted yield means.

CHAPTER 1: GENERAL INTRODUCTION

INTRODUCTION

The corn ethanol industry in the United States has grown over the last 20 years, increasing from 1% to 10% of the total US fuel supply (Renewable Fuels Association 2015). The process has become more efficient, using fewer bushels of corn per gallon of ethanol produced (U.S. Energy Information Administration 2015). On average, ethanol plants produce 2.82 gallons of ethanol per bushel of corn, as compared to 2.51 gallons per bushel in 1994 (Renewable Fuels Association 2015). Much of the industry's current interest centers on maximizing ethanol yield and overall profits. This can be achieved in part by knowing the potential yield of input corn and working to identify what parameters are inhibiting reaching 100% fermentation efficiency. For corn of average composition (71% starch, 9% protein, 4% oil on a dry basis), the theoretical maximum ethanol yield is 2.94 gallons per bushel.

The adoption of rapid and accurate methods of measuring inbound corn quality is not yet widespread in the ethanol industry. Near-infrared spectroscopy (NIRS) can be used to evaluate corn composition and, with an appropriate model, corn composition can be used to predict ethanol yield. Evaluation of predicted ethanol yield versus actual production yield can be used to identify potential for improvement (benchmarking).

There have been attempts to develop models that predict ethanol yield potential based on corn characteristics, such as protein content, starch content, oil content, and kernel density, as determined by NIRS (Hao, Thelen, and Gao 2012; Bryan 2003; Monsanto 2003). Many of these models are held back by real world applicability, in that they were restricted to lab-scale validation, direct NIRS calibrations, or proprietary models/NIRS equipment. The following research was conducted to validate an ethanol yield prediction model equation for dry-grind

ethanol plants, in a commercial setting, using a generic equation intended for all corn hybrids and NIRS equipment.

LITERATURE REVIEW

Dry-Grind Ethanol Production

Approximately 90% of the ethanol industry uses the dry-grind process (Renewable Fuels Association 2015). Dry-grind ethanol plants produce ethanol by breaking down corn starch into simple sugars, and then fermenting those sugars with yeast. The process is shown in Figure 2. Whole corn is ground into flour (most often by hammermills) to which water is added to create a mash. In most plants, the mash is heated and then cooled in the cook step, in order to gelatinize starch and allow enzyme access. One enzyme, alpha-amylase, is added to break up the alpha-1,4 linkages in the starch, reducing it from amylose to maltose and glucose in the liquefaction step. Another enzyme, glucoamylase is then added to finish breaking the starch components into glucose by cleaving alpha-1,4 linkages of non-reducing ends in starch. Glucoamylase also hydrolyzes alpha-1,6 linkages, yielding free glucose (Pavezzi, Gomes, and da Silva 2008). Once starch has been broken into simple sugars, yeast is added to ferment the sugars into alcohol, with a release of carbon dioxide (Figure 1).

$C_6H_{12}O_6$ + H_2O + Yeast →	$2\,CO_2$ +	$2\,C_2H_5OH$ + H_2O +	Heat
Glucose	*Carbon Dioxide*	*Ethanol*	*BTU*
100 lb	48.8 lb	51.1 lb	17,000 BTU

Figure 1: Glucose Fermentaion (Singh et al. 2001)

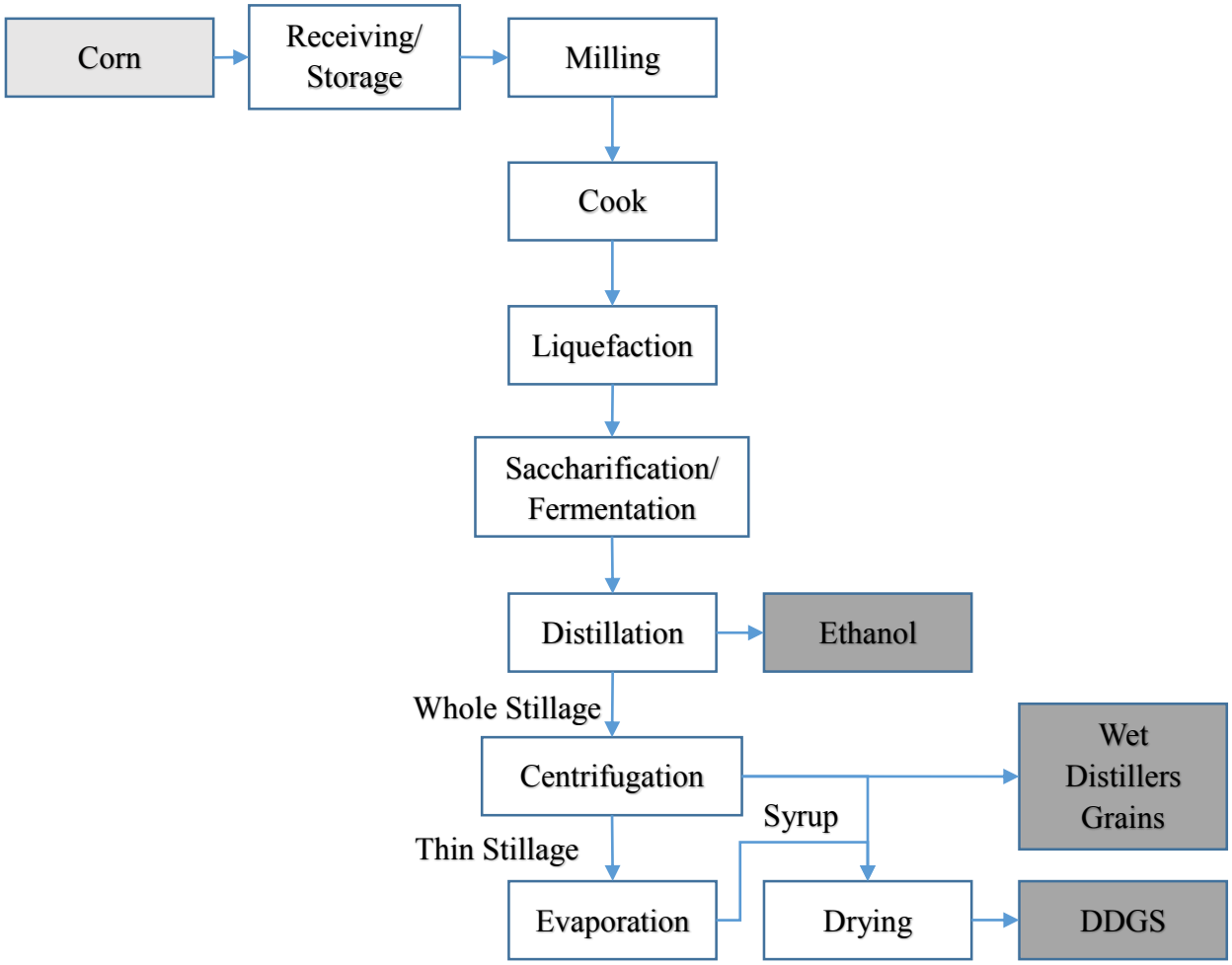

Figure 2: Dry-Grind Ethanol Process

After fermentation, the alcohol is separated from the remaining solids using distillation, then dehydrated, and finally denatured (with regular gasoline) before storage. The solids leave fermentation as whole stillage, which is centrifuged and separated into two streams: thin stillage (low solids) and wet distillers grains, a marketable co-product. Some of the thin stillage is routed through evaporation to be concentrated into a syrup. This syrup can be added back to the wet distillers grains. The mix can be dried to increase shelf-life, which produces the co-product Dried Distillers Grains with Solubles (DDGS). Figure 3 shows the distillers grains marketing choices produced by typical dry-grind ethanol plants. Nearby market availability determines the relative

shares sold by specific plants. Common dry-grind ethanol co-products and their uses can be seen in Table 1.

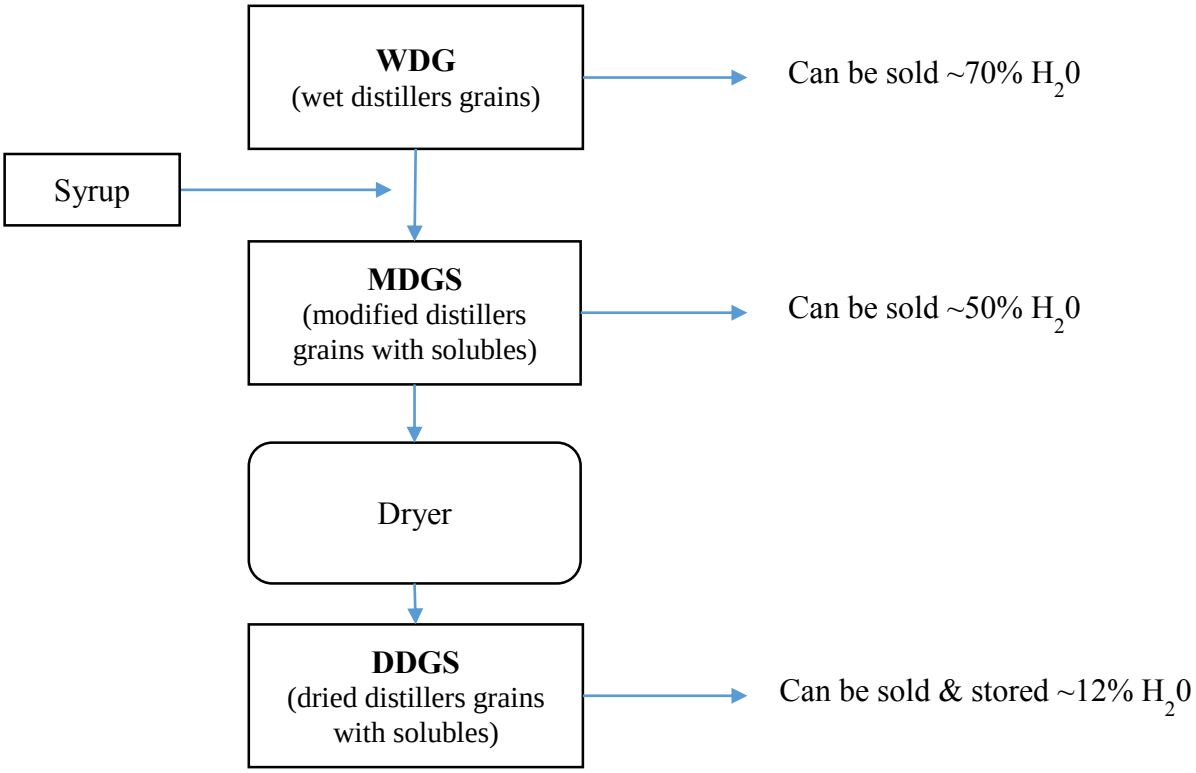

Figure 3: Distillers Marketing Choices

Table 1: Dry-grind ethanol production co-products

Dried Distillers Grains with Solubles (DDGS)	Stillage with syrup added and then dried. Sold as a feed commodity.
Distillers Grains	Stillage that is sold as feed commodity wet or dried.
Corn Oil	Oil extracted from stillage after fermentation. Can be used as an ingredient in biodiesel production.

In a standard dry-grind ethanol plant, one bushel (56 lbs) of corn will produce approximately 2.8 gallons of ethanol (18.1 lbs), 17 lbs of DDGS, and 17.3 lbs CO_2 (Renewable Fuels Association 2015). On average, ethanol plants now produce 2.82 gallons of ethanol per bushel of corn, as compared to 2.51 gallons per bushel in 1994 (Renewable Fuels Association 2015). This increased yield can be attributed in part to better processing efficiency and high fermentable corn

hybrids (Cooper 2015). With the focus on improved starch production and access, corn quality is one of the best indicators of ethanol yield, as the amount of starch determines the theoretical amount of ethanol. Near-infrared spectroscopy (NIRS) can characterize corn quality.

Near-Infrared Spectroscopy

Near-Infrared Spectroscopy (NIRS) is a rapid, non-destructive method of using the near-infrared spectrum to determine the organic composition of a sample (Workman, Jr. 2014). Near-infrared (NIR) energy is a specific region of the electromagnetic spectrum which extends approximately from 780-2500 nanometers (Figure 4). NIRS measures the amount of near-infrared energy absorbed by a sample, which correlates to the sample's chemical compositions.

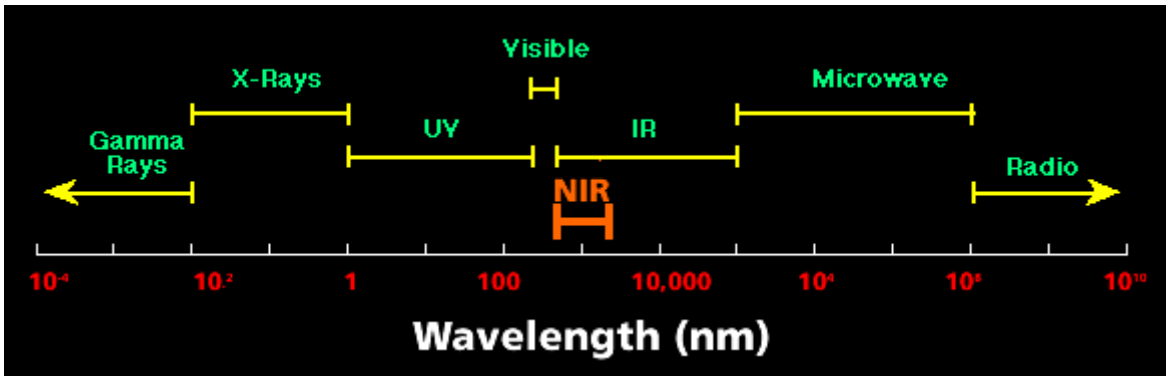

Figure 4: Electromagnetic Spectrum (Analytical 2005)

NIRS analysis is a significantly less time intensive analysis as compared to analytical chemistry, specifically as it requires little to no sample preparation (Davies 2015). The original configuration used diffuse reflectance to measure in the NIR region; now NIRS instruments use either transmittance or reflectance, across a wide array of applications, from agriculture to pharmaceuticals.

NIRS requires the calibration to a set of reference values. Calibrations are then used in the future to compute the composition of samples. These multivariate calibrations quantify the

relationship between instrument spectra and reference data. Reference data, in regards to corn, is laboratory or reference chemistry performed to quantify the grain's composition. Thus, NIRS data is read as the prediction of the sample characteristic in question, for example, protein content. In agriculture, one idea of using NIRS was to "enable detection of quality changes of raw materials and final product under steady process conditions" (Huang et al. 2008).

NIRS can be used to quantify corn composition, both whole kernel and ground samples. Proven models consider moisture, protein, oil, and starch content (%) and kernel density (g/cc). Typical corn composition is made up of 71% starch, 9% protein, 4% oil on a dry basis (Watson 2003). In regards to ethanol production, corn composition indicates ethanol yield, as corn starch is converted to ethanol. The use of NIRS for starch prediction is limited by imprecision in wet chemistry methods used for starch quantification, which provides the reference data for the NIRS calibrations (Hall 2009). Because of the starch measurement limitation, protein is used as the primary indicator of ethanol yield in NIRS. This is appropriate because protein and starch compete in corn kernel grain fill, in an inverse relationship. This means protein and ethanol yield also have an inverse relationship. Complete starch to ethanol conversion is impacted by processes and other production parameters. Monitoring corn quality to establish the theoretical ethanol yield of incoming grain could allow facilities to benchmark parameters that are reducing yield in production.

Current Studies

NIRS has been used to predict ethanol yield. At the commercial level, corporately-created propriety models have been developed. DuPont Pioneer developed a whole-grain Near Infrared Transmission (NIRT) rapid assay (currently known as Pioneer's Ethanol Yield Potential program) to complement its hybrid evaluation program (Bryan 2003). Dry-grind ethanol plants

were able to work with DuPont Pioneer to receive recommended high total fermentable hybrids for given locations. Originally, this work was exclusive to Pioneer hybrids, and was only available for use on Foss Infratec 1241 Grain Analyzer instruments (Bryan 2003). The Illinois Crop Improvement Association independently validated Pioneer's Ethanol Yield Potential calibration in 2008 (DuPont 2008). The propriety calibration, still only for Foss Infratec analyzers, is available through licensing with DuPont Pioneer. In a similar effort, Monsanto's "Fuel Your Profits" program supplied participating ethanol plants with an NIRS instrument to measure a proprietary indicator of corn fermentability at the beginning of the dry-grind ethanol production process (Monsanto 2003). Neither Monsanto nor DuPont Pioneer's products are available outside of company databases, and both are only applicable to Foss Infratec units, which left a need for a more universal method.

Burgers et al. developed a multiple-linear regression equation for predicting corn ethanol yield based on near-infrared spectroscopy (NIRS) measurements of protein, oil, and density on a 15% moisture basis (Burgers, Hurburgh, and Jane 2009). The model equation was intended to be widely applicable, and was validated on a lab scale by the Illinois Crop Improvement Association laboratory. It had the form $y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3$, where β's are regression coefficients for each component (x) of corn composition (protein, oil, kernel density). The original equation was created with crop years and ethanol yields from 2005-2008. Subsequent data was collected from crop years through 2013. The current research updated the model and traced the history of the model over crop years.

An ethanol prediction model is only as good as its real-world applicability. The current study was undertaken to update the Burgers et al. model, then to validate the model in commercial ethanol production facilities. Unlike corporately-developed models, this equation

was intended to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used, as long as the calibration database was consistent. A comparison of the model predicted yield, based on inbound grain composition, and corresponding reported ethanol yield from the same grain was performed to validate the model. The ability of the model to benchmark process control and crop years was assessed. The updated model will reflect, either by long term average or with short term changes, the output of a typical corn dry grind ethanol plant.

THESIS ORGANIZATION

This thesis is organized into two sections. The first section is a general introduction and literature review covering: dry-grind ethanol production, near-infrared spectroscopy, and current studies about predicting ethanol yield using near-infrared spectroscopy. The second part of the thesis is research titled "In-Plant Validation of an Ethanol Yield Prediction Equation." This research involves the commercial validation of a method developed to predict ethanol yield using near-infrared spectroscopy predictions of corn kernel composition. The results from this research are prepared for publication by THE American Association of Cereal Chemists (AACC) in *Cereal Chemistry*.

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CHAPTER 2: IN-PLANT VALIDATION OF AN ETHANOL YIELD PREDICTION EQUATION

A paper to be submitted to *Cereal Chemistry*

Megan Korte and Charles R. Hurburgh Jr.

ABSTRACT

Much of the fuel ethanol industry's current interest centers on maximizing ethanol yield. This can be achieved by knowing the potential yield of input corn and working to identify what parameters are inhibiting reaching 100% fermentation efficiency. Near-infrared spectroscopy (NIRS) is one method that can be used to evaluate corn composition and, with an appropriate model, can be used to predict ethanol yield. Many current models are held back by real world applicability, in that they are restricted to lab-scale validation, direct NIRS calibrations, or proprietary models/equipment. Burgers et al. developed a multiple-linear regression equation for predicting corn ethanol yield based on near-infrared spectroscopy (NIRS) measurements of protein, oil, and density on a 15% moisture basis (Burgers, Hurburgh, and Jane 2009). Unlike corporately-developed models, this equation was intended to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used, as long as the calibration database was consistent. Iterations of the model were evaluated, and the most current version was chosen to use in the rest of the research. A comparison of the model predicted yield, based on inbound grain composition, and corresponding reported ethanol yield was performed to validate the model. On average, the data validated the prediction model. Day to day or batch by batch variability in processing was not accounted for in the equation, but the variability of the corn composition was. From the linear regression analyses performed on each plant, the slopes are the same, but there is a plant-specific bias. The difference between reported and predicted

ethanol yields was negligible (0.0008 gal/bu). Because the equation validated for all plants (RMSE = 0.13 gal/bu), the equation is validated for use, functioning independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used.

INTRODUCTION

The adoption of rapid and accurate methods of measuring corn quality on inbound grain is not yet widespread in the ethanol industry. Near-infrared spectroscopy (NIRS) is a method that can be used to evaluate corn composition and, with an appropriate model, corn composition can be used to predict ethanol yield. Evaluation of predicted ethanol yield versus actual production yield can be used to identify potential for improvement (benchmarking).

There have been attempts to develop models that predict ethanol yield potential based on corn characteristics, such as protein content, starch content, oil content, and kernel density, as determined by NIRS (Hao, Thelen, and Gao 2012; Bryan 2003; Monsanto 2003). Many of these models are held back by real world usefulness, in that they are restricted to lab-scale validation, direct NIRS calibrations, or proprietary models/equipment. The following research was conducted to validate a universal ethanol yield prediction model equation for dry-grind ethanol plants, in a commercial setting.

In a standard dry-grind ethanol plant, one bushel (56 lbs) of corn will produce approximately 2.8 gallons of ethanol (18.1 lbs), 17 lbs of DDGS, and 17.3 lbs CO_2 (Renewable Fuels Association 2015). On average, ethanol plants now produce 2.82 gallons of ethanol per bushel of corn, as compared to 2.51 gallons per bushel in 1994 (Renewable Fuels Association 2015). This increased yield can be attributed in part to better processing efficiency and high fermentable corn hybrids (Cooper 2015). With the focus on improved starch production and access, corn quality is one of the best indicators of ethanol yield, as the amount of starch determines the theoretical amount of ethanol. Near-infrared spectroscopy (NIRS) can characterize corn quality.

Near-Infrared Spectroscopy (NIRS) is a rapid, non-destructive method of using the near-infrared spectrum to determine the organic composition of a sample (Workman, Jr. 2014). Near-

infrared (NIR) energy is a specific region of the electromagnetic spectrum which extends approximately from 780-2500 nanometers. NIRS requires the calibration to a set of reference values, which are then used to compute the composition of the sample. These multivariate calibrations quantify the relationship between instrument spectra and reference data. NIRS data is read as the prediction of the sample characteristic in question, for example, protein content.

NIRS can be used to quantify corn composition, both whole kernel and ground samples. Proven models can determine moisture, protein, oil, and starch content (%) and kernel density (g/cc). Typical corn composition is 71% starch, 9% protein, 4% oil on a dry basis (60.4%, 7.7%, 3.4% on a 15% moisture basis) (Watson 2003). In regards to ethanol production, corn composition indicates ethanol yield, as corn starch is converted to ethanol. The use of NIRS for starch prediction is limited by imprecision in wet chemistry methods used for starch quantification, which provides the reference data for the NIRS calibrations (Hall 2009). Because of the starch measurement limitation, the other components are used as the primary indicator of ethanol yield in NIRS. This is appropriate because protein and oil compete with starch in corn kernel grain fill, in an inverse relationship. Density indicates packing, and has a positive relationship. Complete starch to ethanol conversion is impacted by processes and other production parameters. Monitoring corn quality to establish the theoretical ethanol yield of incoming grain could allow facilities to benchmark parameters that are reducing yield in production.

NIRS has been used to predict ethanol yield. At the commercial level, corporately-produced propriety models have been developed, most notably by DuPont Pioneer and Monsanto (Bryan 2003; Monsanto 2003). Neither Monsanto nor DuPont Pioneer’s products are available

outside of company databases, and both are only applicable to Foss Infratec units, which left a need for a more universal method.

Burgers et al. developed a multiple-linear regression equation for predicting corn ethanol yield based on near-infrared spectroscopy (NIRS) measurements of protein, oil, and density on a 15% moisture basis (Burgers, Hurburgh, and Jane 2009). The Burgers equation was intended to be widely applicable to predict ethanol yield, and was validated on a lab scale by the Illinois Crop Improvement Association laboratory. It had the form $y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3$, where β's are regression coefficients for each component (x) of corn composition (protein, oil, kernel density). The original equation was created with crop years and ethanol yields from 2005-2008. Subsequent laboratory data was collected to include crop years through 2013.

An ethanol prediction model is only as good as its real-world applicability. The current study was undertaken to update the Burgers et al. model, then to validate the model in commercial ethanol production facilities. Unlike corporately-developed models, this equation was intended to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used.

MATERIALS AND METHODS

Model Equation

The previous equation for predicting corn ethanol yield, in gallons per bushel, was developed in 2008. Since 2008, additional data was collected to enable the update of the equation, which is a multiple linear combination of near infrared measurements for corn protein, oil, and density. Starch was not included, because the original research found that the 8 best combinations of protein, oil, starch, and density were not significantly different from one

another, and that starch had a less reliable effect than the other components, due to the reproducibility of the reference laboratory method (Burgers, Hurburgh, and Jane 2009).

The next step in the confirmation of this equation was to update the equation coefficients from the original Burgers et al model:

$$Ethanol\ Yield\ \left(\frac{gal}{bu}\right) = 3.23 - 0.0624 * Protein(\%) - 0.0296 * Oil(\%) + 0.1040 * Density(\frac{g}{cc}) \quad (1)$$

The original model was developed with 246 corn samples from the Illinois Crop Improvement Association. Subsequent iterations using diverse sample sources included normal Iowa corn and inbred/specialty samples (high oil) not normally seen by the ethanol industry. The latest iteration included crop years through 2013. The final version chosen after testing was used for all subsequent yield predictions in this research. The model was developed at the lab scale, with the reference lab being the Illinois Crop Improvement Association.

The most recent iteration of the model had an increased standard error from its previous iteration (0.057 and 0.046, respectively). It included some diverse samples and, when adjusted to mimic the range of protein values seen in commercial practice, was the best version to use for this research. This final version was then used in ethanol plant trials.

Industrial Ethanol Plant Production Trials

Industrial ethanol production plant trials were conducted in four dry-grind ethanol plants, located in Minnesota, Iowa, and Missouri. Plants were numbered 1, 2, 3, and 4 to preserve confidentiality. Rated production capacities ranged from 35 to 70 million gallons per year of ethanol production. Trials began in December 2014 or January 2015, and continued until March 2015. Among these sites, two brands of flow-through near-infrared spectroscopy (NIRS) instruments were used to analyze incoming whole kernel corn moisture, protein, oil, and kernel density. All four NIRS instruments were calibrated by the Iowa State University Grain Quality

Lab to help control instrument variability. Those facilities not previously using NIRS instruments were trained by ISU GQL employees on an NIRS unit provided by Iowa State University. The table below shows characteristics of each plant.

Table 2: Ethanol Plant Characteristics

Plant	Location	Instrument	Trial Start	Corn Source	Sampling Frequency
1	Iowa	Perten 9500*	December, 2014	Truck	1x/batch
2	Iowa	Foss Infratec 1241*	January, 2015	Truck	3x/day
3	Minnesota	Foss Infratec 1241**	January, 2015	Truck	4x/day run 3x each
4	Missouri	Foss Infratec 1241**	January, 2015	Co-op	1x/day (transfer) Run 3x

*owned by company but calibrated by Iowa State
**provided by Iowa State

Site visits

Two site visits were made to each facility prior to beginning the trial: the first to discuss the trial with operators and develop individual plant protocols; the second to train operators to use the NIRS instruments and to deliver NIRS instruments to those that did not previously have them. Employees at each facility were asked to record NIRS data (sample ID, moisture, protein, oil, starch, and density) and corresponding sampling information, such as time, date, sampling location in the plant, and which fermenter or batch to which the sample corresponded. Plants were asked to sample whole corn on receipt, and before hammermills (if possible). Table 3 is a summary of the requested sampling plan.

Table 3: Sampling Protocol

Plant	Inbound Sampling	Before Hammermill	Retention Samples to send to ISU each week
1	NIR already taken on all inbound	1x/batch	3 inbound, 2 before hammermill
2	1-2x daily composite sample	NA	5
3	4x/day on weekdays	NA	2
4	1x composite/day	3x/batch	1 inbound, 1-2 before hammermill

Fermentation data was requested, in particular, bushels of corn entering the fermenter and ethanol yield on a batch-by-batch basis or gallons produced per day, or a daily or batch-by-batch ethanol yield in gallons/bushel. It was then asked that plants retain some samples to be sent to the Iowa State University Grain Quality Lab. The whole samples were to be used to verify data from the plant NIRS instruments.

Retention Samples Validation

Plants sent NIRS data and retention samples (samples of whole corn corresponding as closely as possible to the NIRS data) to Iowa State University (ISU) over a 60 day period. At ISU, NIRS data submitted by the plants was standardized to 15% moisture basis. A total of 648 NIRS analysis results for whole kernel corn moisture, protein, oil, and kernel density were received from participating ethanol plants. The number of NIRS analyses received by ISU were not uniform across different plants, or by day in individual plants. Retention samples ranged from plant to plant, some daily composites, some referenced a specific batch. In total, ISU received 136 retention samples. Once received at ISU, samples were kept in a cooler, and then allowed to warm to room temperature before analysis. All retention samples received at ISU were run on a Foss Infratec 1229 Grain Analyzer NIRS instrument, with the same calibrations as the ethanol plants. Moisture, protein, oil, and kernel density on a 15% moisture basis were recorded. The Foss Infratec 1229 was calibrated with the same calibration data set as the instruments calibrated by ISU GQL at the ethanol plants.

Retention sample NIRS data obtained at ISU was matched to NIRS data provided by plants with a goal of achieving as close a correspondence as possible. For example, typically only 1 sample was submitted per plant, but the same plant may have submitted 3 NIRS analyses that day. In this case, the NIRS data obtained at ISU GQL for the 1 retention sample was

matched to all 3 NIRS analyses for the same day. Protein (% at 15% MB) and oil (% at 15% MB) for retention samples and plant NIRS data were used to validate the NIRS instrument data being taken by each plant. Density NIRS data was not included in the validation, as protein and oil were the largest contributors of corn composition. Statistical analysis was performed using JMP Pro 11.0.0 (SAS Institute Inc., Cary, NC).

Validation of Ethanol Yield Model

After validating the plant-supplied NIRS data with the retention samples, the plant NIRS data was used in the validation of the ethanol yield model. All predicted ethanol yields were predicted using the updated ethanol yield prediction equation:

$$Ethanol\ Yield\ \left(\frac{gal}{bu}\right) = 2.83 - 0.0611 * Protein(\%) - 0.0701 * Oil(\%) + 0.5256 * Density(\frac{g}{cc}) \quad (2)$$

Plant-supplied NIRS data was used in the equation to calculate a predicted ethanol yield. Predicted ethanol yields were compared to plant-reported ethanol yields. Reported yields from plants were all standardized to gallons of ethanol produced per bushel of corn (gal/bu) at 15% moisture, in order to compare them to predicted yields from NIRS data. When possible, predicted and reported yields were matched by batch (as reported), otherwise yields were matched as well as possible on a daily basis. All NIRS data for a day (multiple predicted yields) were matched to that day's reported ethanol yield. If more than one yield was reported for a day, those values were averaged then related to predicted ethanol yields. Plant 4 was unable to supply reported ethanol yield data that could be standardized to gallons of ethanol per bushel corn ground, and subsequently had to be excluded from all analysis.

Reported and predicted ethanol yields for each plant were compared, then fit with a linear regression model. Averages of reported and predicted ethanol yields for each plant were evaluated with corresponding standard deviations. The model was evaluated against predicted

yields for all plants combined, and then for each plant individually. Contrast tests were performed to test the significance of each parameter. Corn composition as reported by plants in NIRS results was also evaluated between reported and predicted ethanol yields.

RESULTS

Updated Model Equation

To determine the best iteration of the validation equation to use for the current research, a test file was developed using a set of normal corn from both a set of strip plots and a set of specialty corn. The progressive model history including number of samples, equation coefficients, and standard error of cross validation, is shown in Table 4, below. The development software was Unscrambler 9.8 (Camo Software AS, Oslo, Norway).

Table 4: Model Development Progression

Model		n	B0	B1*Protein	B2*Oil	B3*Density	SECV
0	Original Equation	237	3.20	-0.0659	-0.0197	0.1290	0.044
1	Burgers Final	293	3.23	-0.0624	-0.0296	0.1040	0.042
2	2009 Model	287	3.14	-0.0624	-0.0529	0.2380	0.031
3	March 2012 Model	396	2.89	-0.0618	-0.0403	0.3930	0.047
4	January 2013 Model	438	2.80	-0.0629	-0.0561	0.5210	0.046
5	February 2014 Model	464	2.83	-0.0611	-0.0701	0.5256	0.057

These regression coefficients are all logical as protein and oil are inverse indicators of yield (due to the inverse relationship with starch). Density should have a positive coefficient, as it is essentially kernel packing, and with protein already accounted for, the packing is the amount of starch in the kernel. More starch would indicate a higher ethanol yield.

The final iteration of the model equation including all sample data was evaluated. A reduced sample set that reflected normal protein content seen at ethanol facilities was tested for robustness. This was done by predicting ethanol yields for the full sample set and the reduced sample set and comparing the standard errors. Table 5 shows the full set and reduced set data.

Average ethanol yields, both predicted by the equation and the corresponding reference yield with standard deviations are shown beside the number of samples for each set. Ranges for protein, oil, and kernel density are also displayed. The reduced set composition ranges more accurately depict what would be received at a commercial ethanol plant.

Table 5: Final Model

	Ethanol Yield	Average (gal/bu)	SD	n	Protein (% on 15% MB)	Oil (% on 15% MB)	Density (g/cc)
Reduced Sample Set	Predicted	2.74	0.12	361	4.0-9.0	2.6-11.5	1.148-1.322
	Reference	2.75	0.09	361			
Full Sample Set	Predicted	2.69	0.20	469	4.0-12.9	2.6-12.7	1.106-1.328
	Reference	2.70	0.16	469			

The standard error of cross-validation of the full model was 0.059 gal/bu. With the reduced range of samples, standard error of prediction was 0.048 gal/bu. The robustness of the center of the model did not change as more variable samples were added (Figure 5). The latest iteration (5), of the model, was used for the plant study.

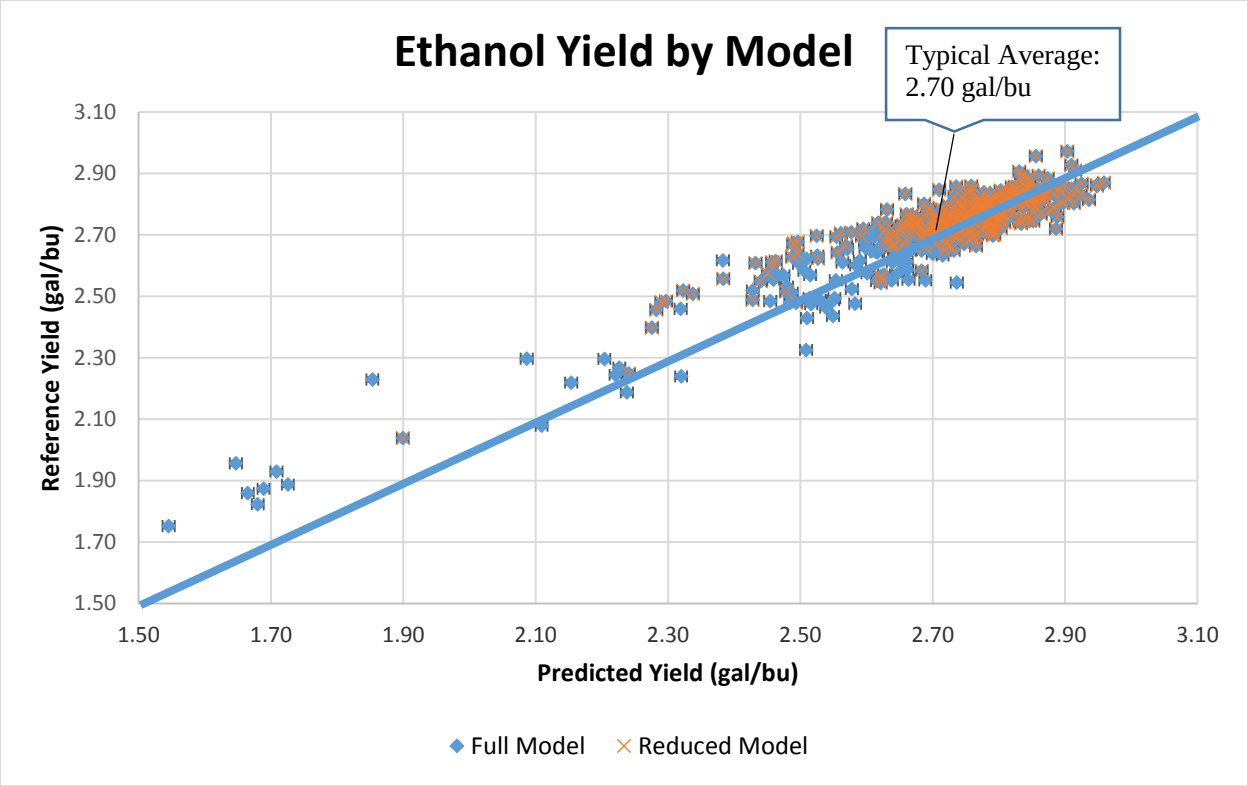

Figure 5: Ethanol Yield as predicted by full and reduced models

Retention Sample Validation

Retention samples sent to ISU were run through a Foss Infratec 1229 Grain Analyzer to obtain NIRS data for protein, oil, and kernel density at 15% moisture basis. The protein and oil NIRS data from the retention samples at ISU were compared to the protein and oil NIRS data received from the plants. Distributions for protein and oil from the facilities and ISU were evaluated. Because individual samples from each source could not be directly matched, a simple linear regression analysis was not performed. Instead, the overall sample set from each source was compared to one another. Sample sets were also evaluated by plant. Protein and oil content of each NIRS analysis was evaluated by two factor Principal Component Analysis (PCA). Grouped by sample source (ISU or plant) did not show significant differences (clustering) between sample source (Figure 6).

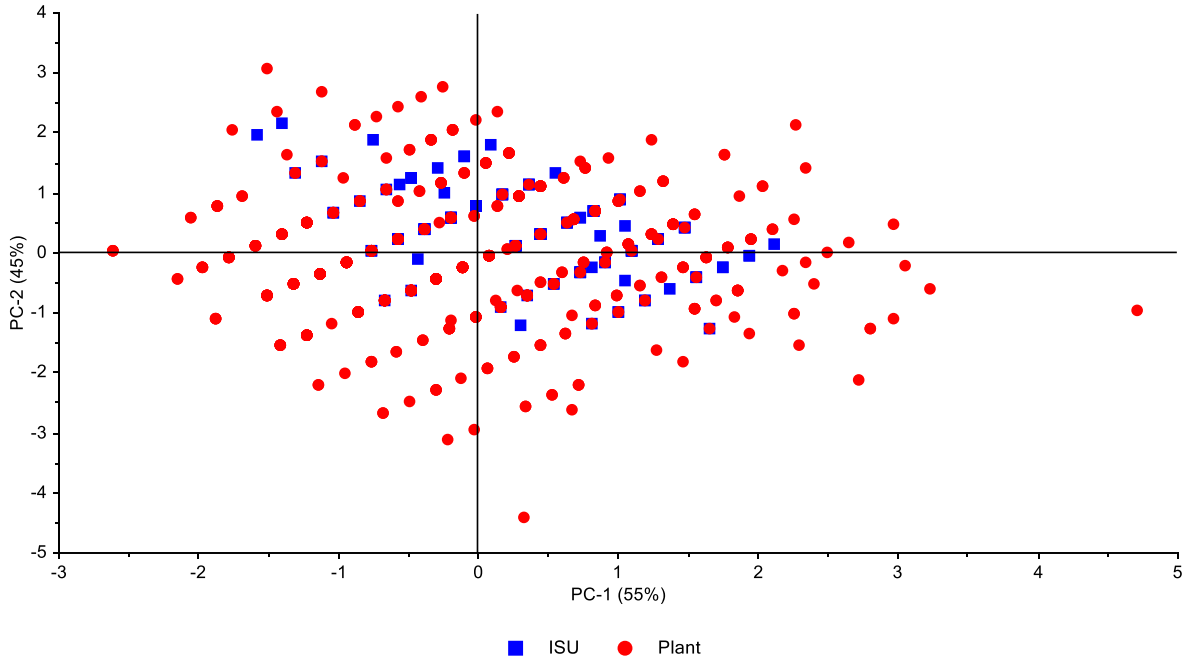

Figure 6: Protein and Oil PCA Analysis of Sample Source

Protein and oil NIRS data from retention samples and plant-reported data were not significantly different (P=0.82 and P=0.32, respectively) as evaluated by ANOVA. The interaction of plant and sample source (NIR analysis of retention sample at ISU or NIR analysis supplied by plant) was signficant for protein, but only for Plant 1 (P<0.0001). The difference between the means from each source was relatively minor at 0.24%. The sample source was not significant for Plants 2 or 3 (P=0.11 and P=0.14, respectively). The table below shows the significance test for source of protein NIRS data by plant.

Table 6: Protein source significance test. Levels not connected by the same letter are statistically significantly different.

LEVEL					MEAN
PLANT 1	A				**7.3**
ISU 1			C		**7.1**
PLANT 2	A	B			**7.2**
ISU 2		B			**7.2**
PLANT 3				D	**6.7**
ISU 3		B	C	D	**6.9**

The interaction of sample source and plant was significant for oil for Plants 1 and 2 (P <0.0001 and P=0.0020), but the interaction was not significant for Plant 3 (P=0.23). Although statistically significant, the difference in the mean oil obtained by sample source (either retention sample analyzed at ISU or plant-supplied data) was relatively minor, at 0.10% pts for Plant 1 and 0.08% pts for Plant 2. The table below shows the significance test for source of oil NIRS data by plant.

Table 7: Oil source significance test. Levels not connected by the same letter are statistically significantly different.

LEVEL					MEAN
PLANT 1				D	**3.3**
ISU 1			C		**3.4**
PLANT 2	A				**3.5**
ISU 2		B			**3.5**
PLANT 3			C		**3.4**
ISU 3			C	D	**3.3**

Confirming that the source of the data (ISU or plant-supplied) was not significant for either protein or oil validated the plant-supplied NIRS analyses, and allowed it to be used in the validation of the ethanol yield model.

Validation of Ethanol Yield Model

Ethanol yields were predicted using the validated, plant-supplied NIRS data as inputs in the updated equation (Model 5, Table 4). Predicted yields were compared to reported yields from each plant in Table 8.

Table 8: Plant Ethanol Yields

		Min (gal/bu)	Max (gal/bu)	Ave (gal/bu)	SD (gal/bu)
Plant 1	Predicted	2.77	2.85	2.81	0.02
	Reported	1.98	3.09	2.77	0.15
Plant 2	Predicted	2.77	2.85	2.81	0.02
	Reported	2.50	2.82	2.71	0.06
Plant 3	Predicted	2.72	2.90	2.85	0.02
	Reported	2.10	3.18	2.86	0.19

There were significant differences among the plants in the study between reported and predicted yields ($P<0.0001$). A linear regression analysis was performed.

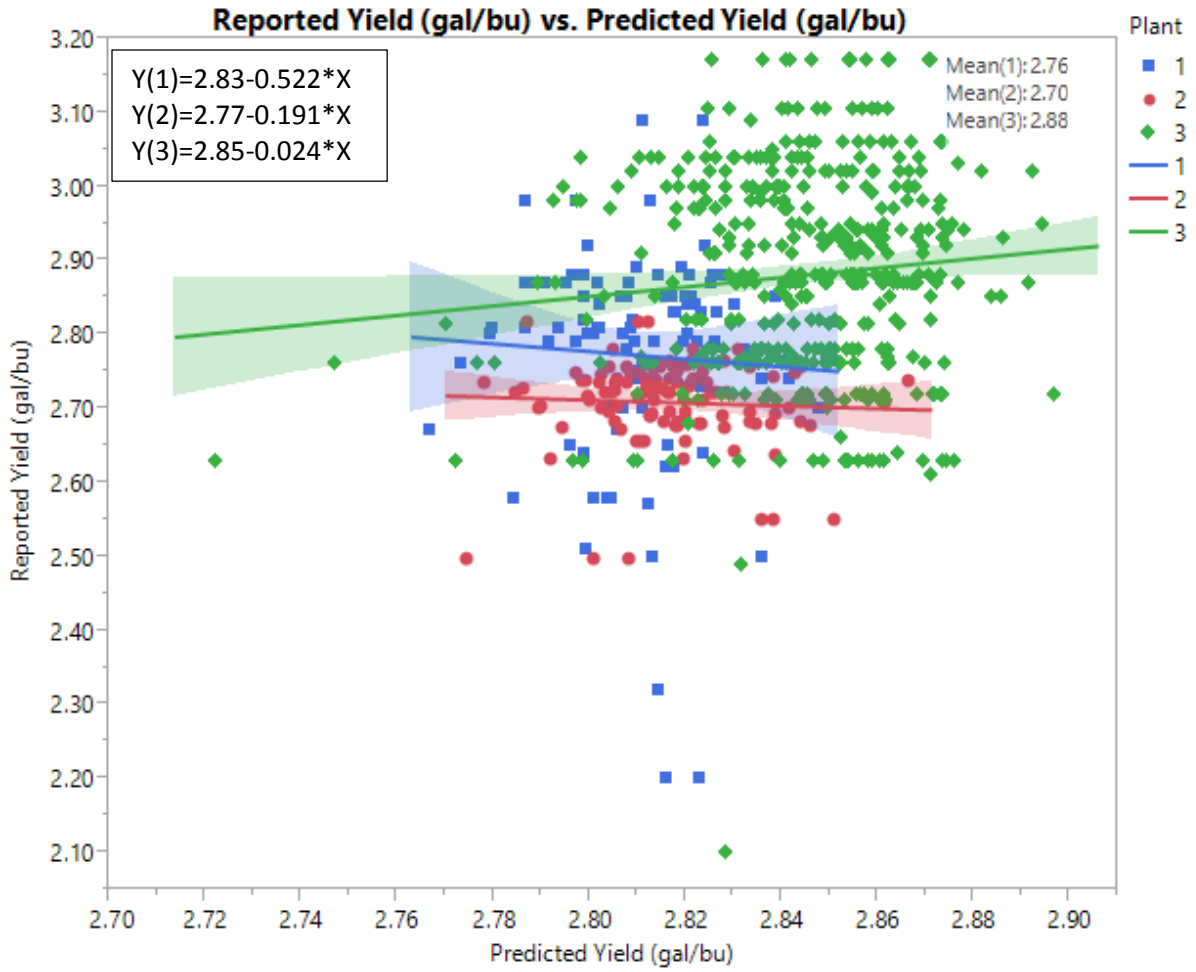

Figure 7: Reported vs Predicted Ethanol Yield Regression Analysis by Plant

The slopes for the three plants' predicted and reported ethanol yields did not differ significantly from one another. Plant 1 and 2 slopes did not differ significantly from 1 (P=0.09 and P=0.12). The slope of Plant 3, however, did differ significantly from 1 (P=0.01). The intercepts of Plant 2 and Plant 3 differed (P<0.0001), while the intercepts of Plants 1 and 3 did not differ significantly (P=0.27). Overall, the combined model for the linear regression produced a low R^2 value (0.23) which shows that a significant amount of variability in the data is not explained by the model.

Plant-supplied NIRS data were used as inputs to generate predicted ethanol yields using the updated equation. Predicted ethanol yields were then compared to facility-reported yields for

each plant (Figure 8-10). Each graph shows the predicted ethanol yield in gallons per bushel, from the plant-supplied NIRS data, the plant-reported ethanol yield, and the averages of the two ethanol yields over the trial period. These figures show the magnitude of differences between predicted yield from corn composition and reported yield from the plants. There is a large variation in reported yields, with substantial maximums and minimums. These discrepancies could be data reporting issues or time series errors in data recording. Some of the largest reported yields were greater than even the theoretical maximum yield, clearly showing data reporting discrepancies. For two of the three plants (1 and 3), the average of the predicted and reported yields are nearly identical. For Plant 2, there was a 0.10 gal/bu consistent difference.

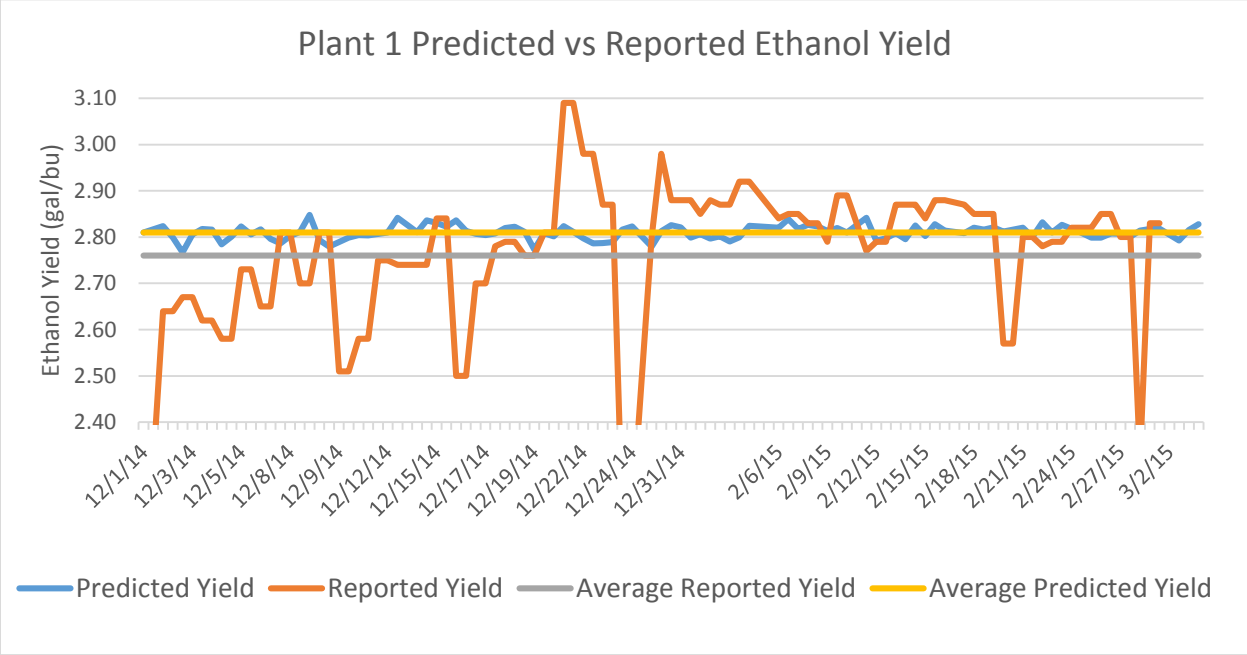

Figure 8: Plant 1 Reported and Predicted Yields During Trial Period

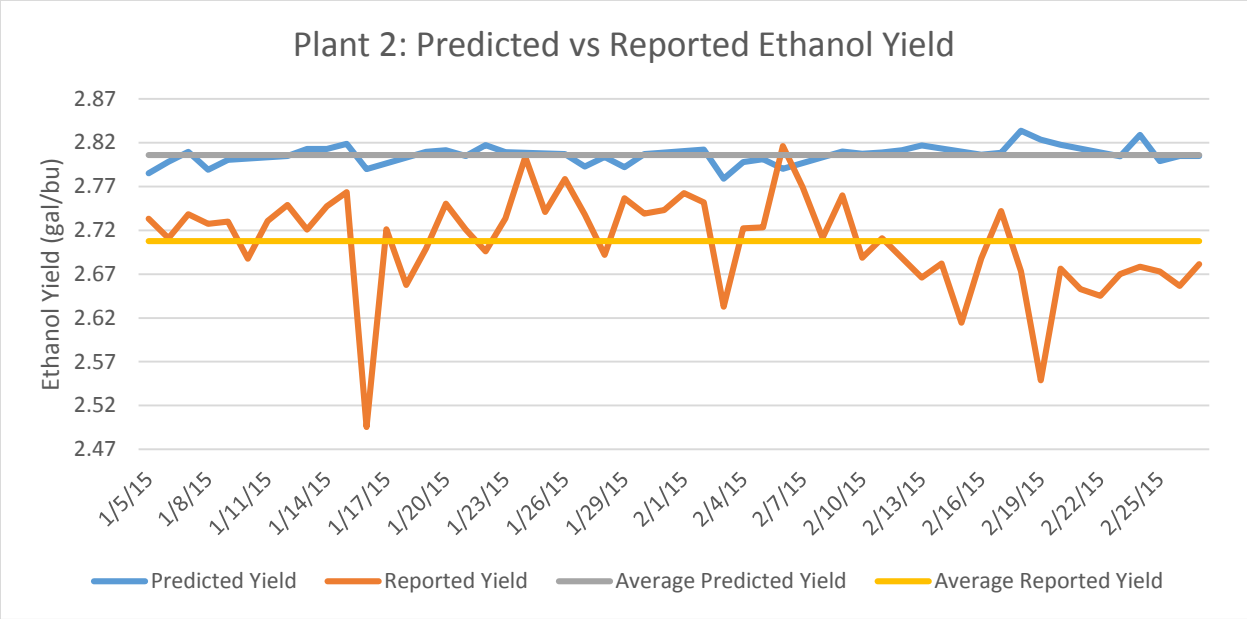

Figure 9: Plant 2 Reported and Predicted Yields During Trial Period

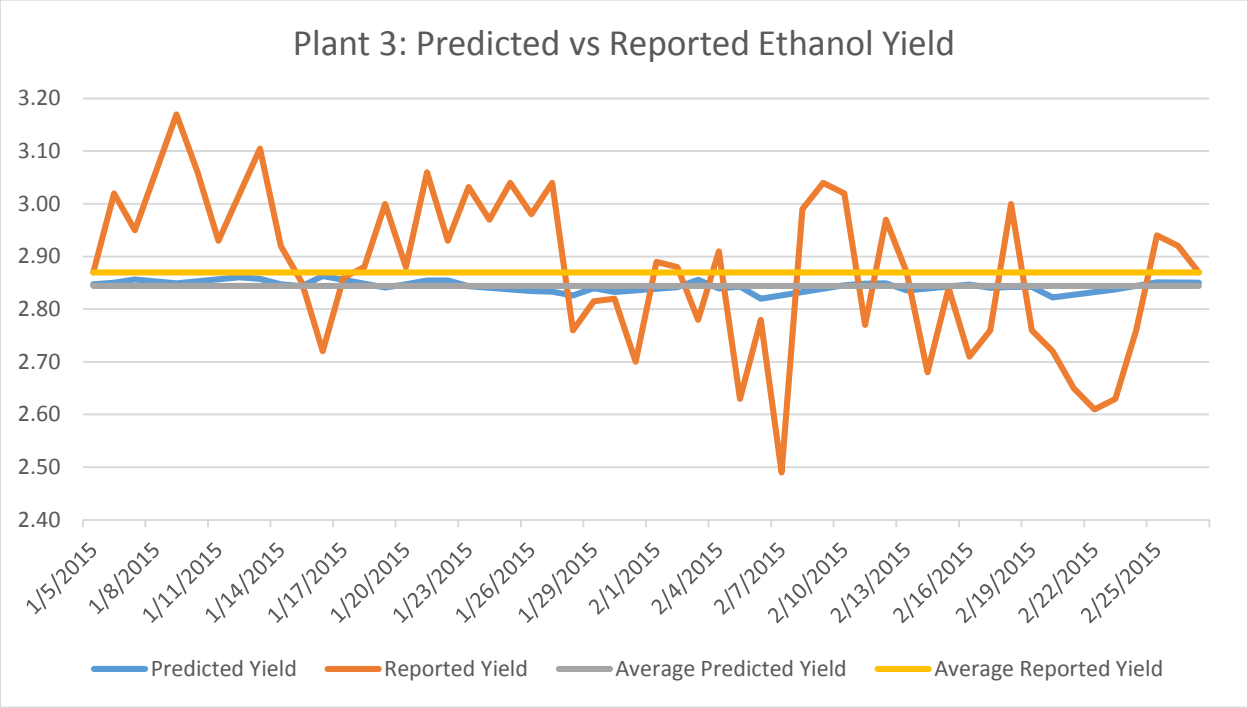

Figure 10: Plant 3 Reported and Predicted Yields During Trial Period

In order to evaluate response of the model with regards to corn composition variability, versus other, unknown variability, ethanol yields (predicted and reported) were compared to NIRS protein content values. Corn kernel protein content is one of the best indicators of ethanol yield, inversely when evaluating corn by NIRS. As can be seen in Figure 11, protein and predicted ethanol yield vary inversely, with an overall R^2 value of 0.79, while the correlation between protein and reported ethanol yield does not have such a clear relationship, and an overall R^2 value of 0.27.

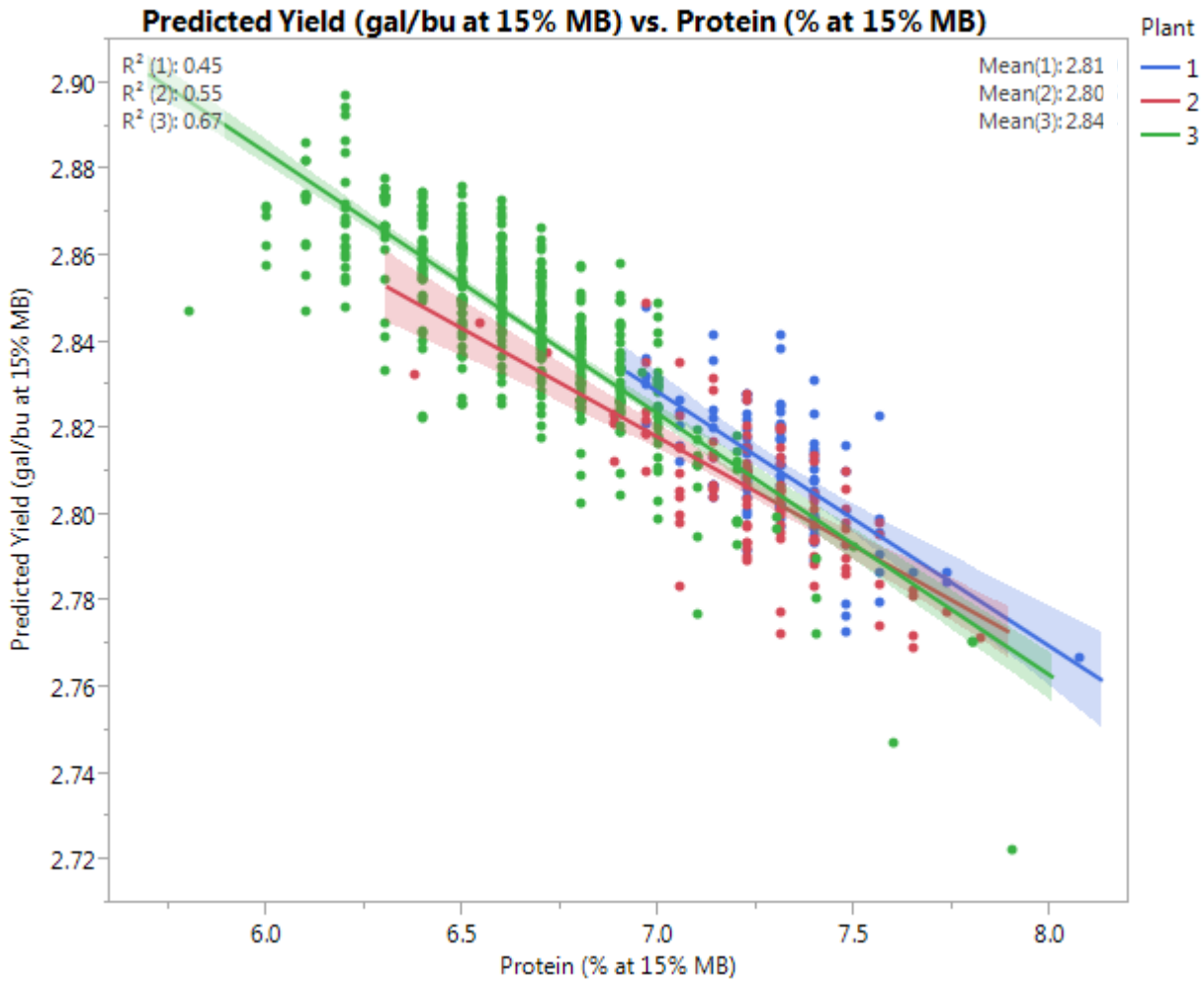

Figure 11: Predicted Ethanol Yield vs Protein Content Linear Regression

Figure 7 shows the ability of the model to predict ethanol yield, but also shows that there is variability that cannot be predicted from a model based only on corn composition. The protein versus the predicted ethanol yield shows a clear inverse relationship, which is the expected relationship between protein and ethanol production (Figure 11).

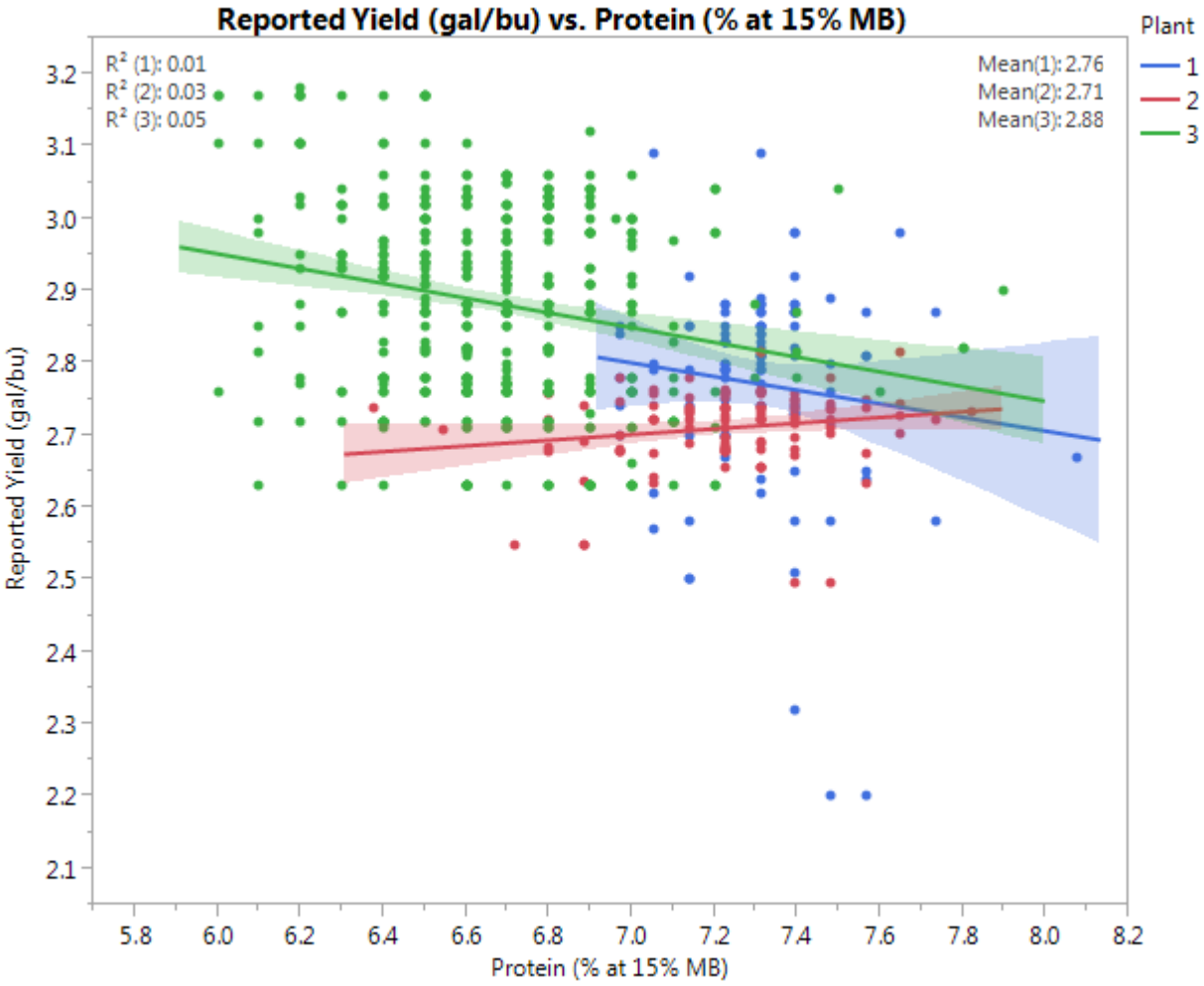

Figure 12: Reported Ethanol Yield vs Protein Content Linear Regression

The protein as compared to the reported ethanol yields does not show that clear relationship (Figure 12). This shows that there is something else affecting ethanol yield in the production facilities. Because corn composition can be ruled out from the model, it may be

inferred that there is processing variability, and likely data, that is altering the reported yield as compared to the predicted yield.

DISCUSSION

The difference between NIRS data at ISU from the physical retention samples and the NIRS data sent from the plants was not significant, taking into consideration that data could not be matched perfectly to retention samples. Average protein readings were 0.2 (% protein) different, with standard deviations of 0.17 and 0.18 (% protein) for plant-reported NIRS data and NIRS data obtained at ISU, respectively. The difference between the protein data was considered insignificant for this research, as it results in only a 0.01-0.02 gal/bu difference when used in the model (Version 5), well within its standard error. There was no difference in the means of oil data between plant and ISU analyses. Evaluating the NIRS data supplied by the plants with physical retention samples analyzed by ISU GQL validated the plant-supplied analyses, allowing them to be used in the rest of the research more confidently.

On average, the data validated the prediction model. Day to day or batch by batch variability in processing was not accounted for in the equation, but the variability of the corn composition was. From the linear regression analyses performed on each plant, the slopes are the same, but there is a plant-specific bias. Figure 13 shows the averages of the predicted and reported yields by plant for the trial period. The predicted yields are close, yet the reported yields are noticeably different for each facility. Processing or data management differences could be the cause for these differences, which supports the idea of a site-specific bias for the equation. Management at a facility could use the bias as a benchmark parameter to meet by improving ethanol yields (such as Plant 2 with a 0.10 gal/bu offset).

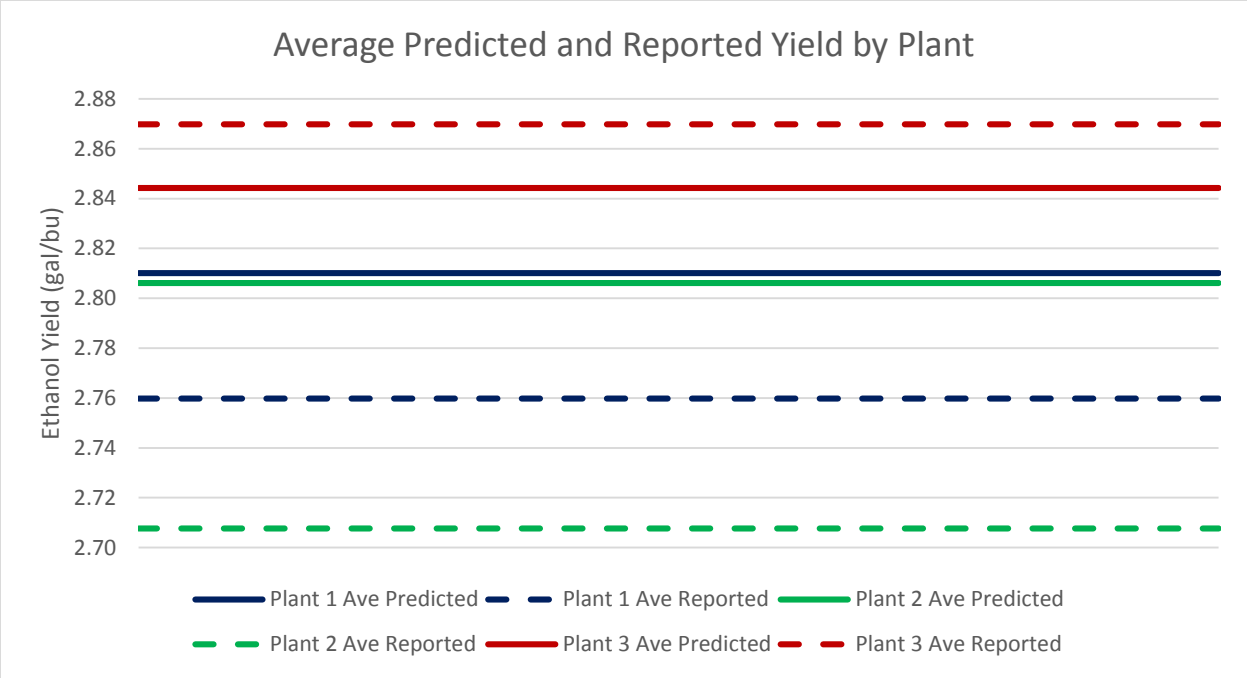

Figure 13: Average Predicted and Reported Yields by Plants during Trial Period

Benchmarking is a way to quantify success or shortfalls of production on commercial scale. Because the equation predicts ethanol yield well for plants on average, it can be used to benchmark process controls across crop years. For example, crop year changeover at ethanol plants can be a time of processing inconsistencies, especially if corn composition changes significantly. Figure 14 shows year by year data of corn protein content (15% MB) for 7 locations in Iowa (Nelson 2015). From 2012 to 2013 there is a change in protein content from approximately 8.7% to 6.7% in the same county (Blackhawk). With all other components of the equation kept constant, the predicted ethanol yield difference would be approximately 0.10 gal/bu. During the first few weeks of harvest season, unexpected variations in output are typical; origination, in part, is from uncontrolled mixtures of dissimilar crop years' corn.

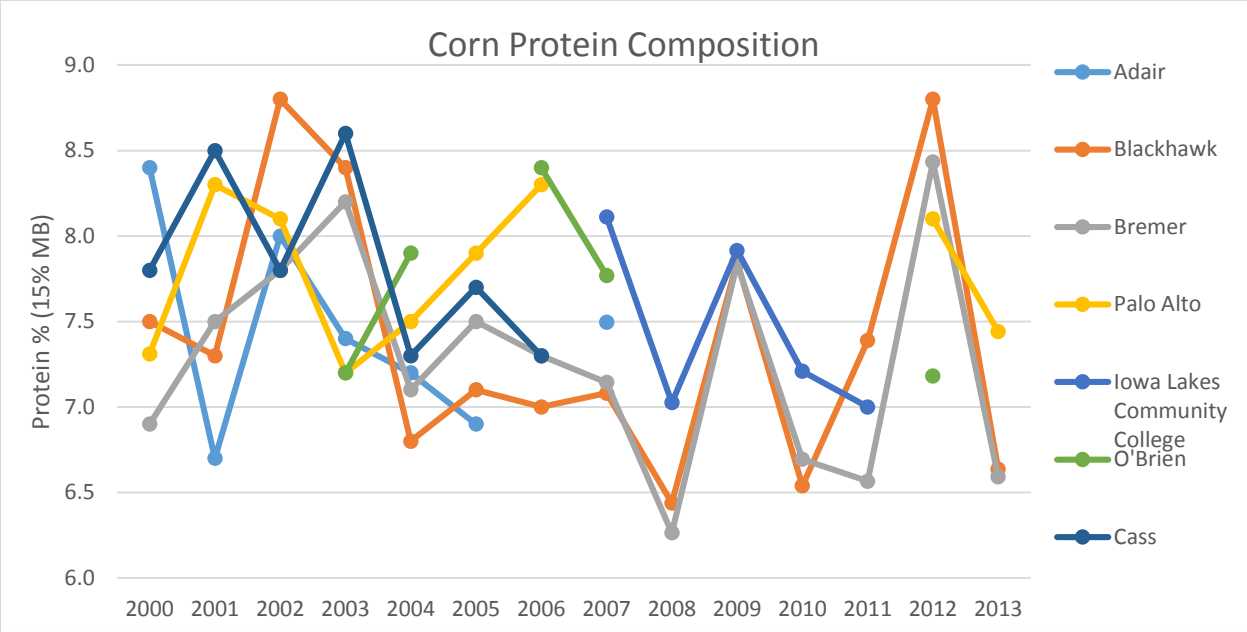

Figure 14: Corn Protein Composition for 7 locations in Iowa from 2000-2013

Major changes in process control can be monitored with this equation within the same crop year, when corn composition is (reasonably) constant. To use the model, plants would need to identify key areas causing the most variability and implement mitigations for those first, and then move on to smaller and smaller causes of variability. For instance, plants could focus on fermentation times/ temperature ranges, then work down to screen size on hammermills or enzyme dosing. Crop year changeover can also be monitored, specifically when corn composition changes significantly from year to year. Monitoring these quality differences can allow for planning and process controls to mitigate the effects of variable corn composition. This would be a first step in developing inbound corn quality management protocol. Again for example, plants may find that corn quality is, on average, different in one part of their trade area versus another.

One inhibitor of this research was a lack of tracking of grain through storage and into processing by plants. If sites were to encourage better tracking through storage, a more accurate

correlation between inbound grain and output ethanol could probably be made, rather than an average. The ability of a plant to manage the process this closely is unknown, however.

CONCLUSION

The 2008 equation was updated over five iterations, progressively including data from 2009-2013 crop years. This equation identified key corn quality parameters. Because the equation validated for all three plants, the equation is validated to function independently of corn hybrid, corn supplier, growing location, and NIRS instrument make/model used. Different corn hybrids were supplied to different plants, and with the different plant locations, corn supplier and growing locations were varied. Two NIRS make/models were used across the plants, and the data was validated by a third make/model at the Iowa Grain Quality Lab at Iowa State University. The model validated with a root mean square error of 0.13 gal/bu, and no difference (0.0008 gal/bu) between overall reported and predicted yield means. This signifies that the model is valid for use by commercial facilities to predict ethanol yield from corn composition.

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CHAPTER 3: GENERAL CONCLUSIONS AND RECOMMENDATIONS

The model can effectively show what the plant output is likely to be, based on the incoming parameters. Refining traceability through a facility would allow for matching inbound corn, to corn entering the process after storage, to what corn specifically is in a particular fermentation batch. Knowing average corn composition and corresponding average theoretical yield is a great first step in encouraging tracking. Encouraging sampling for inbound grain and before the hammermills would provide a means to monitor on-site storage and degradation concerns. Focusing on traceability would be a quality control parameter to optimize yield and improve processing that does not require any major equipment or process changes.

One of the ways the model could be used would be to combine it with other models to evaluate co-products. The change in ethanol yield affects the amount and composition of co-products. Facilities could charge a premium to guarantee certain compositions of co-products, while knowing ethanol yield production and being able to plan around that at the same time.

The model can also be a benchmarking tool. Identifying and quantifying key performance indicators, starting with this equation would significantly increase quality control abilities. This would begin with the development of a comprehensive inbound corn quality management protocol. Knowing theoretical yield of a batch or even just on a daily average would begin to highlight processing deficiencies, especially if the deficiency was consistent over time. Implementing this model on-site would be a first step in maximizing yields and, in turn, plant profitability. Clearly, the variation in plant reported yields indicates either plant process or data collection issues exist.