Use of FT-NIR Spectroscopy in Fuel Ethanol Industry

Introduction

Ethanol is a renewable fuel made from a variety of plant feedstocks, grains and starches. Since the United States has an abundant supply of corn (its number one crop), most of the ethanol produced in the US is made from corn. The Clear Air Act (1970/1990) mandates the addition of oxygenates (such as ethanol) to lower carbon monoxide emissions, reducing air pollution. Utilizing ethanol as a fuel additive reduces greenhouse gas emissions by up to 59 percent relative to gasoline. According to the U.S. Energy Information Administration, weekly ethanol production averaged 1.02 billion barrels per day during the first six months of 2017; up 5% from the same period in 2016. The U.S. Department of Energy states that nearly 97% of gasoline containing ethanol is classified as E10 (10% ethanol, 90% gasoline), which requires no special fueling equipment, and can be used in any conventional gasoline engine.

Ethanol production has steadily increased since the 1980s and continues to grow as the U.S. works to reduce its dependency on foreign energy supplies. As such, quality control has become a top priority for ethanol producers. Process improvements is areas such as dry milling, fermentation, and distillation are being implemented throughout the industry.

In the past decade, near infrared (NIR) spectroscopy has been successfully utilized in the fuel ethanol industry in the following principal areas: monitoring the quality of incoming grains; fermentation process monitoring; distillate analysis; and blending and dry house operation. Compared with traditional methods, such as wet chemistry and HPLC, NIR spectroscopy provides considerable advantages in fuel ethanol quality control applications, such as:

- Eliminates costly consumables such as solvents, columns, and reagents
- Provides extremely fast results; generally less than 1 minute measurement time per sample
- Allows simultaneous analysis of multiple components per sample
- Effectively eliminates sample preparation time
- Eliminates many sources of systematic errors due to sample preparation and instrument misuse

FT-NIR Spectroscopy for the Fuel Ethanol Industry

Incoming Grain

Weather conditions contribute greatly to the quality of corn produce used in ethanol production. For example, frost before the crop is fully matured can end the grain-filling process. Excessive rainfall and hail can damage the crop and increase the chance for field mold and ear rot. At ethanol producing facilities, corn is accepted or rejected based on the moisture and starch content. NIR spectroscopy provides a rapid, nondestructive analysis method of raw material.

Whole corn can be analyzed by Fourier transform near infrared (FT-NIR) spectroscopy using a diffuse reflectance measurement. Values for moisture, protein, oil, and starch can be obtained in seconds.

Fermentation Process

For ethanol production, it is essential to have a consistent fermentation process. Using FT-NIR specrtoscopy, fermentation can be consistently monitored either online or offline.

Using the online approach, a diffuse reflectance probe is inserted into the fermenter for direct and continuously updated analysis.

To monitor the mash solid mix, the offline method analyzes the sample using an integrating sphere. Parameters such as ethanol content, carbohydrates, acids, and glycerol content can be monitored at different stages of fermentation and compared to the profiled record. Any abnormality can be recognized and corrected immediately.

Real-time process monitoring by NIR spectroscopy can be used to evaluate enzymes, yeasts, and additives used in fermentation and to improve the process yield. Higher ethanol content generated during the fermentation process reduces distillation cost and maximizes profit.

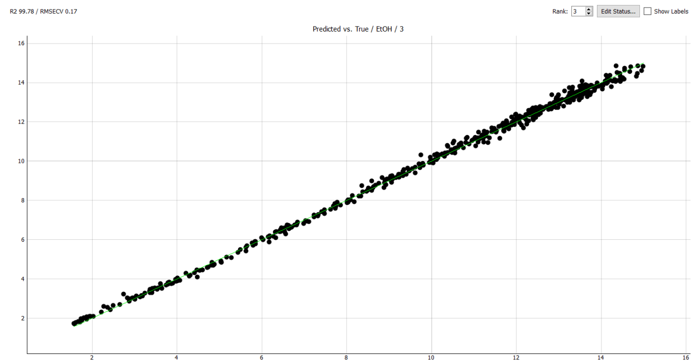

Figure 1: Fermentation of corn mash ethanol, cross-validation result (R^2=99.78, RNSECV=0.17).

Distillate Analysis

After fermentation, the mixture is then put through the process of distillation to separate the ethanol content from water. Because ethanol has a lower boiling point than water, it will vaporize first. The captured vapor is then cooled and compressed into liquid form; FT-NIR analysis of the liquid can be used for quality control purposes.

Figure 2: Distillates in a glass vial analyzed with the QuasIR™ 4000 sample compartment.

Dry Milling Process

There are two main processes for turning corn into ethanol: wet milling and dry milling. In the US, dry milling has become the primary method, largely because it is the most economical. This is partly because one of its byproducts is dried distiller's grain with solubles (DDGS), which is commonly sold as high-energy, high-protein feed for livestock.

Constant quality control is vital for DDGS. Traditional wet chemical methods for monitoring quality parameters can take up to 3 days for analysis. With NIR spectroscopy, DDGS samples can be analyzed within seconds to determine protein, fat, starch, and other nutrients.

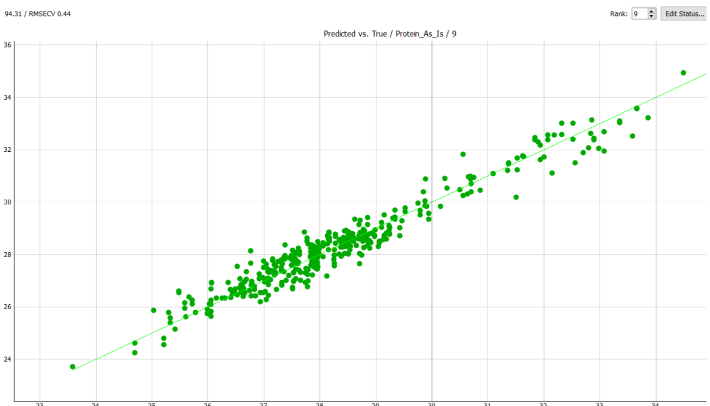

Figure 3: DDGS protein cross-validation result (R^2=94.31, RNSECV=0.44).

Conclusion

FT-NIR provides a rapid, nondestructive method for analyzing ethanol content and other parameters (e.g. moisture, protein, carbohydrates, acids, etc.) in every stage of ethanol production.

The fundamental benefits of FT-NIR analysis in ethanol production include:

- Elimination of costly consumables such as solvents, columns, and reagents
- Provides extremely fast results: generally, less than 1 minute measurement time per sample
- Allows for simultaneous analysis of multiple components per sample
- Effectively eliminates sample preparation time, which is ideal for raw material analysis
- Eliminates many sources of systematic error

Figure 4: Corn sample in rotating cup analyzed with QuasIR™ 4000 integrating sphere.

Galaxy Scientific's QuasIR™ 4000 is ideal for offline analysis of fuel ethanol plant offline analysis. The large sampling area integrating sphere is excellent for inhomogeneous solid samples such as incoming corn and DDGS, or suspension samples such as corn mash. The sample compartment is ideal for transmission measurements of liquids such as distillates.

GS-A-Fuel Ethanol-0.1EN

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