Applications Report
Feed Stock Oil Excerpt

Topic: NIR Analysis of Process Biodiesel Properties (FeedStock)

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Contents:

Study Objective/Summary

The purpose of this study is to report the ability of Verum Analytics' NIR technology to measure key biodiesel properties at various stages in the production process. Four process points were evaluated; Pre-Dry, Post Separation, Post Dry and Glycerin. Properties being reported included Moisture, Free Fatty Acids (FFA), Total Bound Glycerin, Mono and Methanol. Primary values for the constituents of interest were determined by the customer using their standard laboratory methods.

The results demonstrate that LT-NIR effectively measures the desired properties at all stages, from incoming pre-process oils to fished biodiesel, and recovered products such as glycerin. Strong correlations of the LT-NIR measured values to the primary values are shown below for incoming feedstock vegetable oils.

Experimental Procedure

The supplied samples were scanned using an Verum's ParaFuel Spectrometer with an insertion Probe (Figure 1) connected via NIR-grade fiber optic cables. Samples were taken over the range of 1200-2400 nm. Samples were scanned once or twice depending on the process point. Spectra were processed for absorbance using a scan of air as the background/reference.

Figure 1: Analyzer with Transflectance Probe

The scan time for each scan was set to 30 seconds for all cases. Scan time is adjustable, depending on measurement accuracy needs, with longer scan times enabling more accurate (higher signal-to-noise) measurements.

Data Analysis and Results

The full results from the study are presented below in a table for convenience. In the sections that follow, more details are provided for each sample set individually.

Stage	Samples	Property	Range	SEP	R^2-SEP
Pre-Dry	30	FFA (%)	0.05 – 17.5	0.37	0.99
Pre-Dry	30	Moisture (%)	0.1 – 1.5	0.08	0.96
Glycerin	29	Methanol (%)	0.13 – 0.97	0.029	0.98
Post-Separation	35	FFA (%)	0.55 – 1.27	0.07	0.96
Post-Separation	60	Total Bound Glycerin (%)	0.49 – 5.57	0.075	0.99
Post-Dry	47	FFA (%)	0.003 – 0.245	0.007	0.98
Post-Dry	60	Moisture (ppm)	68 - 1159	57	0.98
Post-Dry	60	Total Bound Glycerin (%)	0.139 – 0.724	0.014	0.97
Post-Dry	60	Mono (%)	0.158 – 0.706	0.039	0.96

Table 2: Results Summary

Pre-Dried Feed Stock

Thirty samples of vegetable oil feed stock were supplied with known values of Free Fatty Acid and Moisture. These are the two most common properties of interest to optimize the biodiesel manufacturing process. As shown below the calibration models for FFA and Moisture show positive measurement performance. The standard processing options Mean-Centering and First Derivative were applied for calibration. These and all standard processing options are automatically incorporated into the finished calibration for a fully automated measurement process.

Stage	Samples	Property	Range	SEP	R^2-SEP
Pre-Dry	30	FFA (%)	0.05 – 17.5	0.37	0.99
Pre-Dry	30	Moisture (%)	0.1 – 1.5	0.08	0.96

Table 3: Results for Pre-Dry Samples

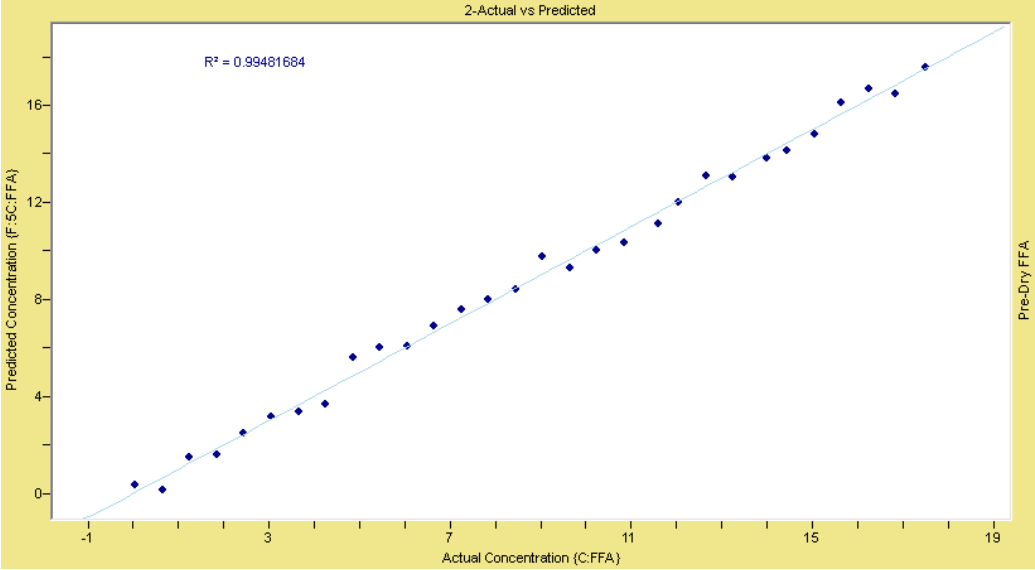

Figure 2: Actual vs. Predicted Values for Free Fatty Acid (FFA) in Pre-Dry Samples

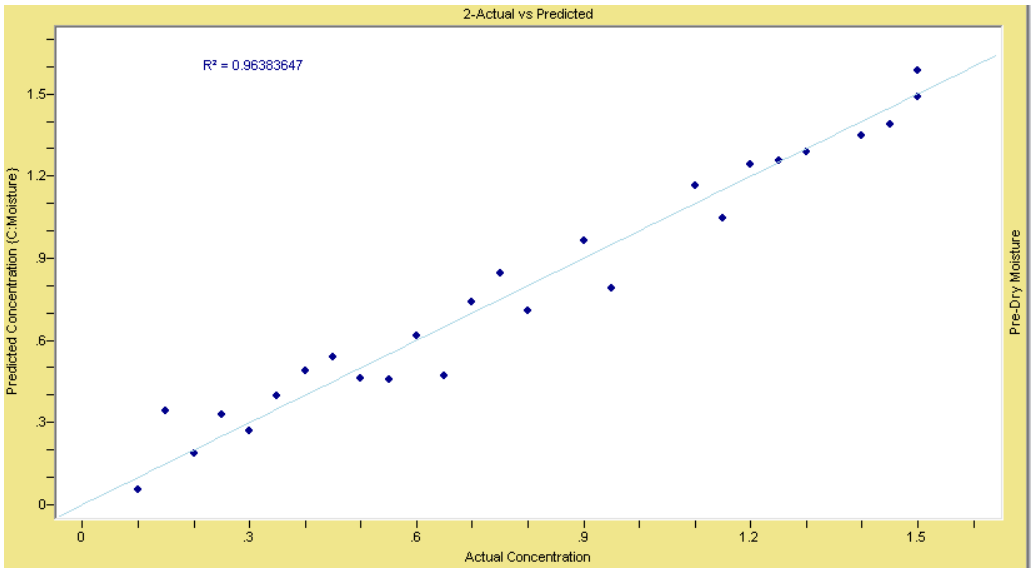

Figure 3: Actual vs. Predicted Values for Moisture in Pre-Dry Samples

Results will be improved upon with a larger sample set. The analyzer would typically measure moisture to accuracies on the order of +/- 0.005%, so we expect that there was variation between the sample measured in the laboratory and the sample being measured by the ParaFuel.

Conclusions and Recommendations

The results of this report indicate that LT-NIR is well suited for online or laboratory measurement throughout the biodiesel production process, as indicated by the good calibration measurement capability for samples in the pre-dry, post-separation, post-dry, and glycerin phases of production. In all cases, good-performing NIR models were developed for the properties of interest. These results provide an indication of the Analyzer's abilities and results can be improved by increasing the quantity, accuracy, and breath of the sample sets.

The LT-NIR technique can be applied inline for direct real-time process monitoring and control or in laboratory measurements. Verum Analytics' online analyzers include a high-performance analyzer, probes and/or flow through cells, multiplexer for measurement of up to 20 process points, fiber cables to connect the analyzer to the probes/cells. The inline analyzer system provides multi-point monitoring and integration of results directly to the process controller (DCS) or data historian system. Verum Analytics will configure the system to meet your specific requirements.

A variety of online implementations are available to suite the specific process or integration requirements. Verum can assist with in-process integration and engineering considerations by providing configuration and technical information as well as budgetary pricing. Please contact Verum Analytics for more information on NIR solutions and configurations for this measurement.

Figure 11: Online ParaFuel Analyzer

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