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Real time monitoring & control of biodiesel/renewable diesel pre-treatment

Key Words

- Renewable diesel pretreatment
- Phosphorous
- FFA
- Trace metals
- Water
- Real time monitoring

Abstract

The static optics IRmadillo has been successfully used to measure contaminants (free fatty acids (FFA), trace metals and phosphorus) in a range of biodiesel feedstocks. The results showcase the IRmadillo's ability to continuously monitor these concentrations in real time, specifically in the pre-treatment phase.

Introduction

Globally, there is a growing focus on moving away from the use of traditional petrochemical and fossil fuel-based energy sources due to their unsustainable, environmentally damaging and non-renewable nature. More sustainable alternatives are biodiesel and renewable diesel, which are produced from triglycerides found in fats, oils and greases. Due to the variety of feedstocks used to produce biodiesel and renewable diesel, a pre-treatment step is essential to remove various contaminants responsible for the fouling of equipment and the deactivation of valuable catalysts in downstream operations. This usually involves various degumming and bleaching processes to remove these impurities. Monitoring the pre-treatment process is therefore paramount to ensuring the process runs efficiently. Some of the impurities measured at this stage are phosphorus (P), metals and free fatty acids (FFA). Traditionally, these are measured off-line using titrations and inductively coupled plasma (ICP) techniques which can be slow and laborious. Continually monitoring these impurities online can result in a better understanding of the process, enabling real time analysis and effective process control. Here we present the use of a static-optics inline FTIR instrument to calibrate for contaminants (P, FFA and various metals) in biodiesel feedstocks. We show that the calibration models are independent of feedstock, making them ideal for biodiesel/renewable production where feedstock types tend to vary.

Experimental

This work was performed on-line using the IRmadillo spectrometer. Spectra were acquired at a resolution of 16 cm-1, where each sample was measured in triplicate and averaged over 120 s. Reference data was supplied by the customers: phosphorus and metals measurements were obtained using ICP and FFA was measured by titration. Models were developed using EVRI's SOLO chemometric modelling software using either locally weighted regression (LWR) or support vector machine regression (SVM-R).

Results and Discussion

The instrument has been installed inline downstream of the washing centrifuges using Keit's proprietary

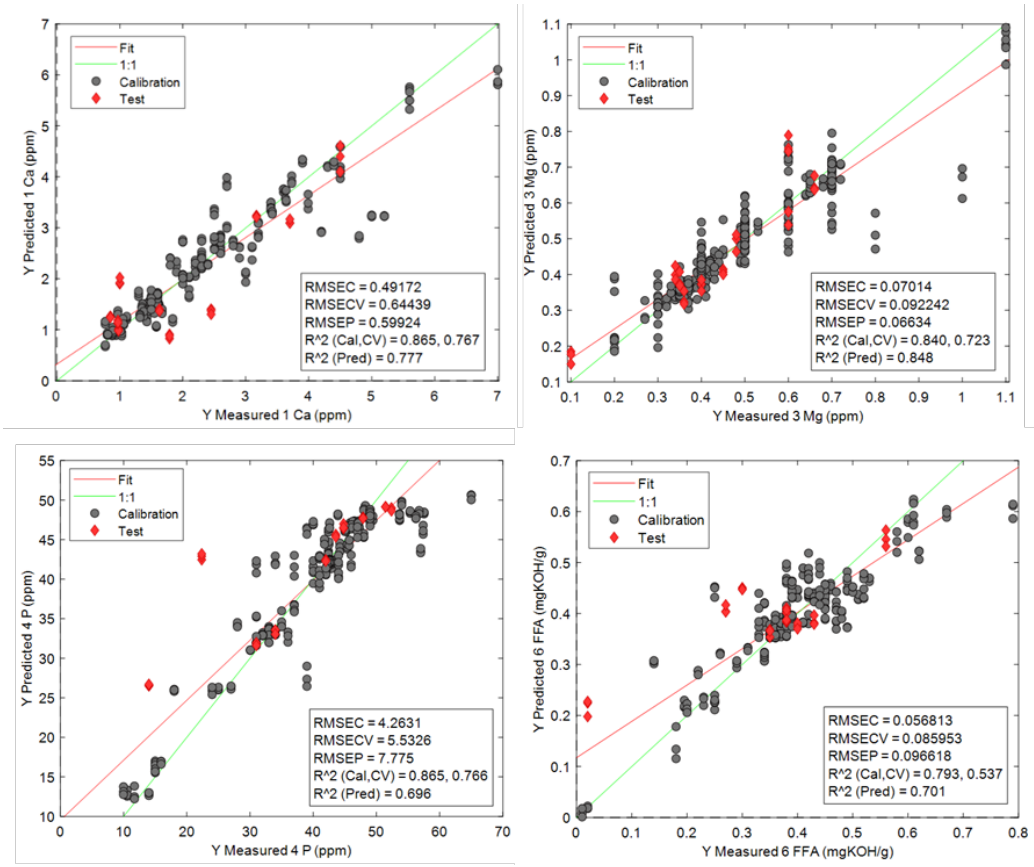

Figure 1: Correlation plots for Ca, Mg, P and FFA

mechanical interface. Each component modelled well showing good agreement between the measured and predicted data as in Figure 1. Models were evaluated based on root mean squared errors of cross validation (RMSECV) and prediction (RMSEP) that represent average error across the dataset, where the RMSEP is evaluated using samples unseen by the model and RMSECV using subsets of the calibration data. These values are shown in Figure 1 where the average errors of cross validation are about 0.09 ppm, 5.53 ppm, 0.64 ppm and 0.09 ppm for FFA, P, Ca and Mg respectively. Correlation plots (measured concentration against

predicted concentration) for each of these species are also shown in Figure 1 where the black dots represent the calibration data, and the red diamonds show the blind predictions. There is good agreement between both measurements for each chemical species.

Feedstocks for biodiesel and renewable diesel can vary considerably and include cooking oil, soybean oil, tallow, chicken fat and beef tallow. Therefore, being able to continuously monitor contaminant levels to the same degree of accuracy using the same chemometric model regardless of feedstock is also important for process efficiency. In the example shown here, the original calibration was built using data obtained in March. Feedstock changes were observed in June where the original models did not perform as well. The model space was then expanded to account for this change making the models more robust to these differences. Figure 2 presents the real time measurements of these updated models overlaid with the corresponding offline ICP/titration measurements for the new feedstock.

Conclusions

This work successfully presents the use of a robust and reliable static optics FTIR to measure impurities in biodiesel feedstock presenting real time measurement in the pre-treatment phase of production, replacing the need for traditional off-line measurement techniques that are unable to provide continuous monitoring and real time process control.

Although the work above focuses on phosphorus, FFA, Ca and Mg other impurities such as total acid number (TAN), other trace metals and nitrogen have also been quantified in renewable diesel feedstocks using the IRmadillo, increasing the value of this application in renewable diesel pre-treatment processes.

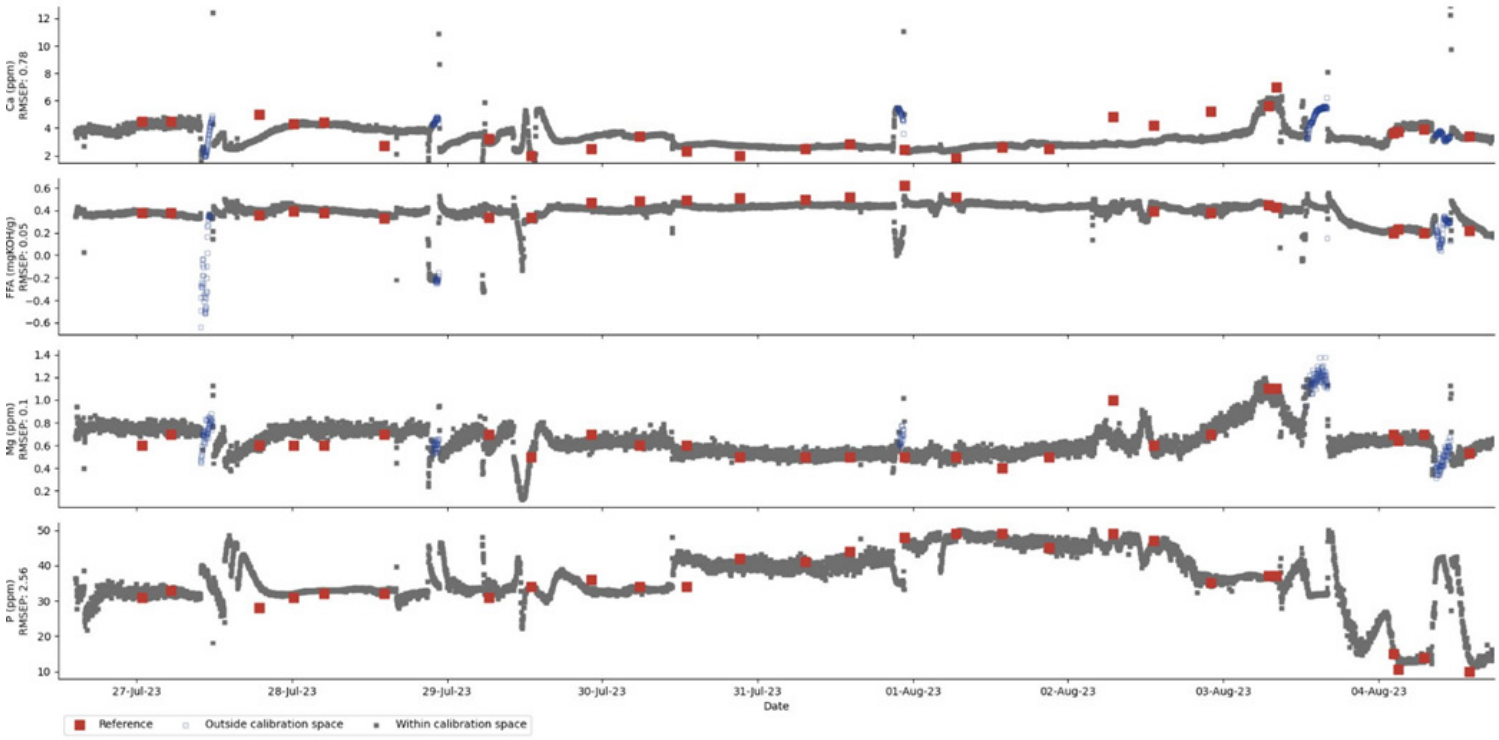

Figure 2: Real time measurements of Ca, Mg, P and FFA

Keep in Mind

Keep in mind the IRmadillo is an FTIR, not an NIR, instrument. There has been a lot of work trying to bring NIR instruments to the manufacturing floor, and various attempts have been made to use NIR for process measurement and control in manufacturing industries. Unfortunately, the fundamental physics behind NIR means it's not an ideal solution for the challenges faced in renewable diesel production, and struggles to provide meaningful information. The IRmadillo is an FTIR, not an NIR, so has substantially more information available for interpretation!

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