Application Note

KEIT spectrometers

Real-time monitoring of **FFAs & FAMEs during edible oil refining process** with an in-line process analyser

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Abstract

There is a need in the edible oil industry to accurately monitor components such as free fatty acids (FFAs) and fatty acid methyl esters (FAMEs) to accurately dose the process for alkali neutralisation. Current technologies struggle to accurately monitor these components in real time. Here we successfully demonstrate how the Keit IRmadillo FTIR spectrometer can provide real-time, in-line analysis of FFAs and FAMEs thereby making it a powerful monitoring solution in the processing of edible oils.

Introduction

Refining of edible oils is a key part of the food supply chain, with many different steps and processes. These range from simple filtration steps through to complex enzymatic or chemical reactions. Monitoring these processes in-line remains a challenge, but the benefits of doing so can be significant: 'What you don't measure, you can't control'.

Laboratory experiments using mid-infrared spectroscopy (FTIR) are well known, but conventional process FTIR instruments make use of fragile fibre cables and sensitive arrays of moving mirrors – making them wholly unsuitable for refinery use.

Through the use of static optics and a solid "light pipe" approach to a probe, we have created an instrument that yields the information quality of an FTIR spectrometer, but with dramatically improved robustness and reliability for real-time results: the IRmadillo.

Here we present our results of how the IRmadillo process analyser can be used to successfully measure FFAs and FAMEs in oil and enable real-time control of the alkali refining process.

IRmadillo™

Key Words

- Edible oil
- Alkali refining
- Free fatty acid (FFA)
- Fatty acid methyl ester (FAME)
- Process control
- In-line process monitoring

Features & Benefits

- Mid-infrared/FTIR spectral analysis
- Vibration tolerant
- Long-term stability
- Low maintenance
- Compact design
- Real-time, multi-component analysis
- Easy to use

Experimental

A mixture of free fatty acids (FFAs) and fatty acid methyl esters (FAMEs) in sunflower oil was prepared using a design of experiment (DoE) methodology. These samples were analysed on the IRmadilloDiamond spectrometer for 120 s with three repeats.

The spectra were then analysed using Camo Analytics Unscrambler 10.5, with a standard normal variate transform (to normalise the spectra) followed by support vector machine regression (SVR) modelling. This technique was used due to the non-linear correlation between the components to be measured and the spectral response.

Results and Discussion

The spectra are shown in Figure 1 with various features of interest highlighted. These band assignments are as follows:

a) C=O in FFAs

b) C=C bonds in unsaturated FFAs and FAMEs

c) C-H bends in FFAs and FAMEs

d) C-C and C-O stretches in FFAs and FAMEs

e) O-C=O bend in FFAs

f) 0-C=O bends in FFAs and FAMEs

The relatively small differences in spectra are easy to see with an FTIR (mid-infrared) instrument, but are extremely difficult to observe and analyse using near-infrared (NIR) instrumentation.

These peaks are used in chemometric models to form calibration curves, and the entire spectrum is interpreted simultaneously for enhanced accuracy and precision.

SVR calibration models for FFAs and FAMEs dissolved in sunflower oil are shown in Figure 2. The average error across this concentration range (which is used as a limit of detection – LoD) is 0.24% for FFA and 0.06% for FAME.

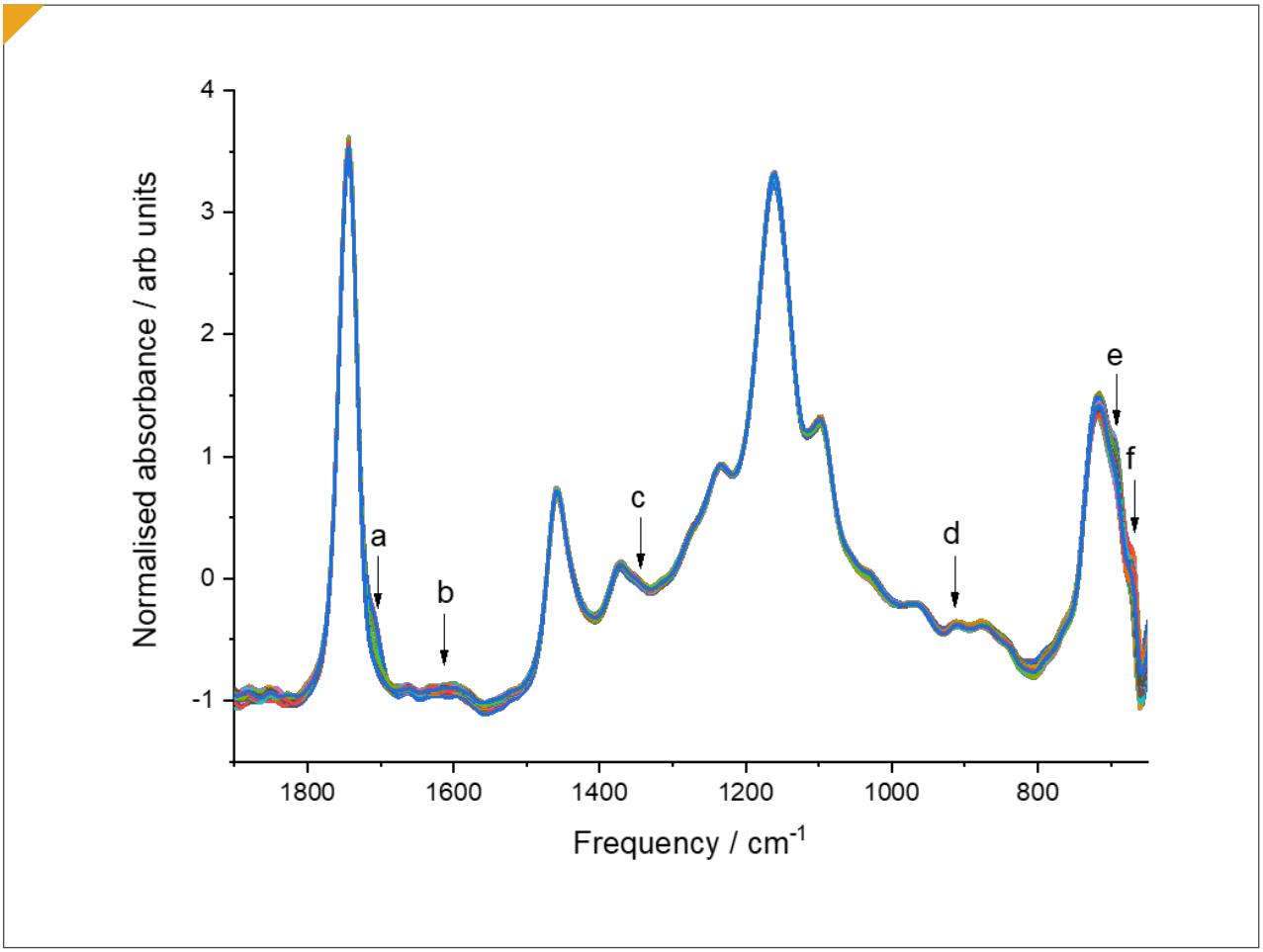

Figure 1: Spectra acquired on the IRmadillo during calibration of FFA and FAME mixtures in sunflower oil.

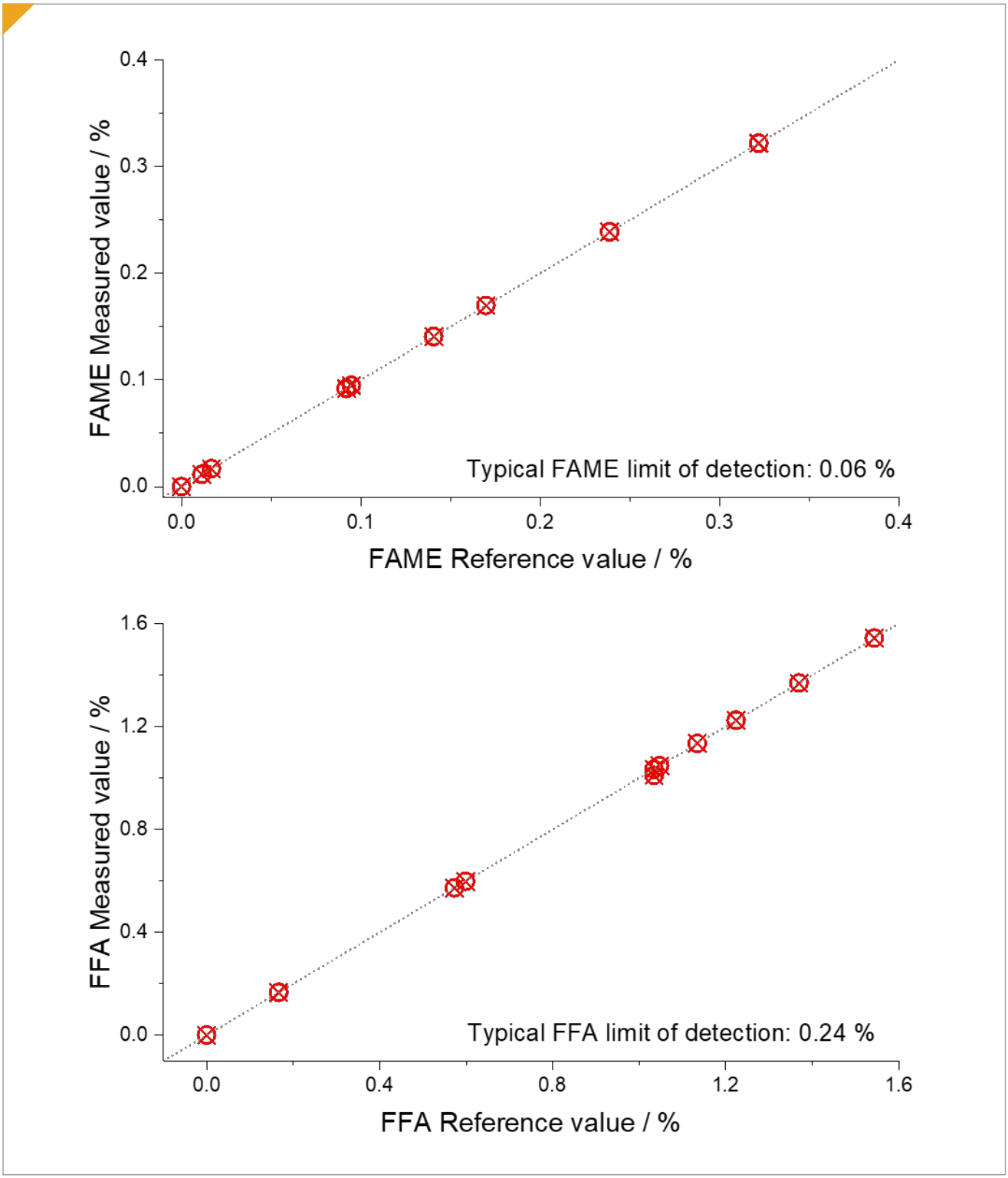

Figure 2: Calibration curves for FFAs and FAMEs dissolved in sunflower oil.

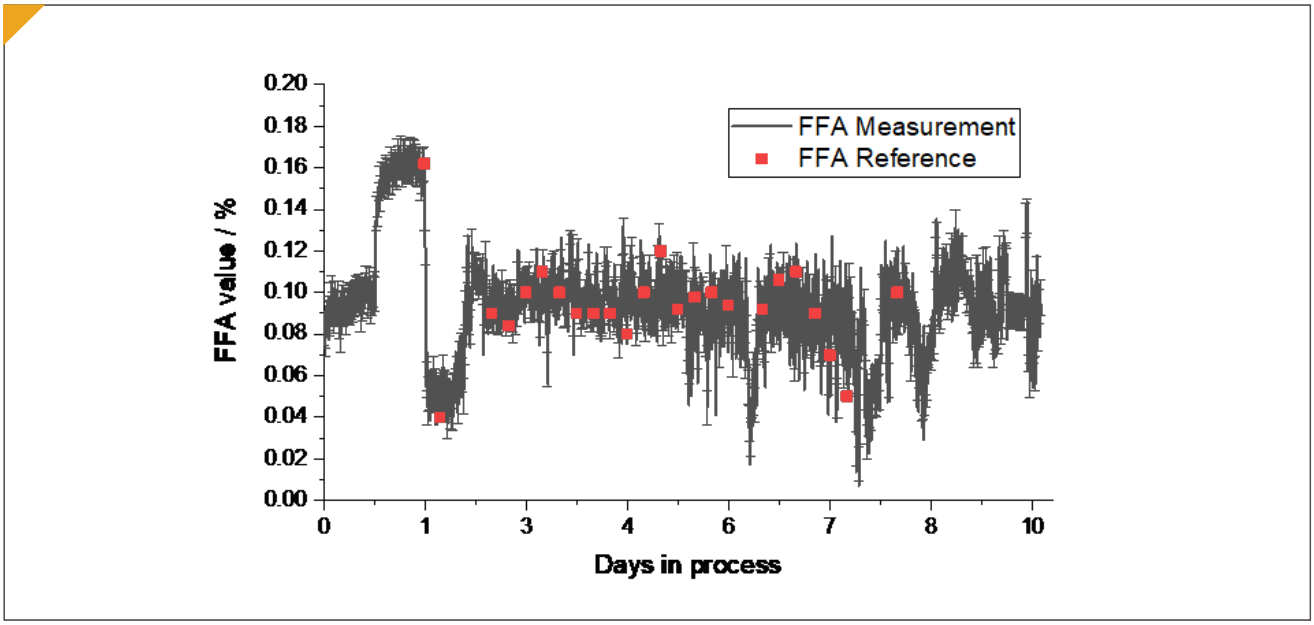

Figure 3: Trace of FFA values over time from IRmadillo installed in an edible oil refinery over a ten-day period.

10-day in-Line monitoring of FFAs

An IRmadilloDiamond instrument has also been installed in-line in an oil refinery at the end of the refining process to ensure the FFA neutralisation is effective. This refinery processes multiple oil types and a single calibration was built for all types.

The results of a 10-day processing period are shown in Figure 3. The IRmadillo is in excellent agreement with the reference values (lab values), and also shows both the natural gentle variation over time as well as large fluctuations in processing and oil quality.

For example, there is a clear increase in FFA value at 0.5 days which remained in the process for a further 0.5 days before it was identified by off-line titration (shown with the reference value). This was quickly rectified in the refinery and the IRmadillo captures the exact moment the alkali dosing neutralised the oil.

The IRmadillo also highlights that the oil quality for the whole of day 1 was purer than necessary – potentially costing the refinery additional money in alkali cost.

Conclusions

The IRmadillo has been shown to be an effective analyser for FFA monitoring in edible oil refining. It has uses throughout the edible oil refining process, especially in the control of chemical dosing (both caustic/lye and phosphoric acid), as well as oil quality checking towards the end of the process line.

The IRmadillo can be calibrated to monitor many different chemicals in the process at once, especially (but not limited to):

- Free glycerol
- Water
- Free fatty acids (FFAs)
- Fatty acid methyl esters (FAMEs)
- Phospholipids
- Soaps
- Chlorophyll
- Iodine value (IV)

IRmadillo™

Keep in mind

The IRmadillo is not a near infrared (NIR) instrument. The IRmadillo is a process analyser based on FTIR (mid-infrared) spectroscopy.

What does this mean for you?

It means the level of information the IRmadillo provides is much more detailed than NIR, enabling better analysis, less calibration and more informative analysis!

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