

## Key Words

- Edible oil refining
- Free fatty acids
- FFA
- Neutralization
- NaOH control
- Caustic control
- NIR
- FTIR
- Spectroscopy

## Introduction

Traditional approaches for measuring free fatty acid (FFA) in crude and bleached oils involve taking a sample to the laboratory for titration testing. This is a laborious process, and only informs the operators what the FFA content was when the sample was taken rather than after the titration has been performed. Furthermore, titrations are an expensive use of resource, as they are slow and difficult to perform.

Spectrometers allow continuous and detailed measurement of chemical concentrations in real time. Unfortunately the majority of process spectrometers are based on near infrared light, which is fundamentally less informative than mid infrared light. Conventional mid infrared spectrometers (which often use a Fourier transform are referred to as "FTIR spectrometers") have sensitive moving parts and fragile fibre probes - making them wholly unsuitable for production environments such as oil refineries.

The IRmadillo is a process analyser built using FTIR spectroscopy, but with static optics: removing the need for moving mirrors or fibre probes, and dramatically improving stability, reliability, and ruggedness.

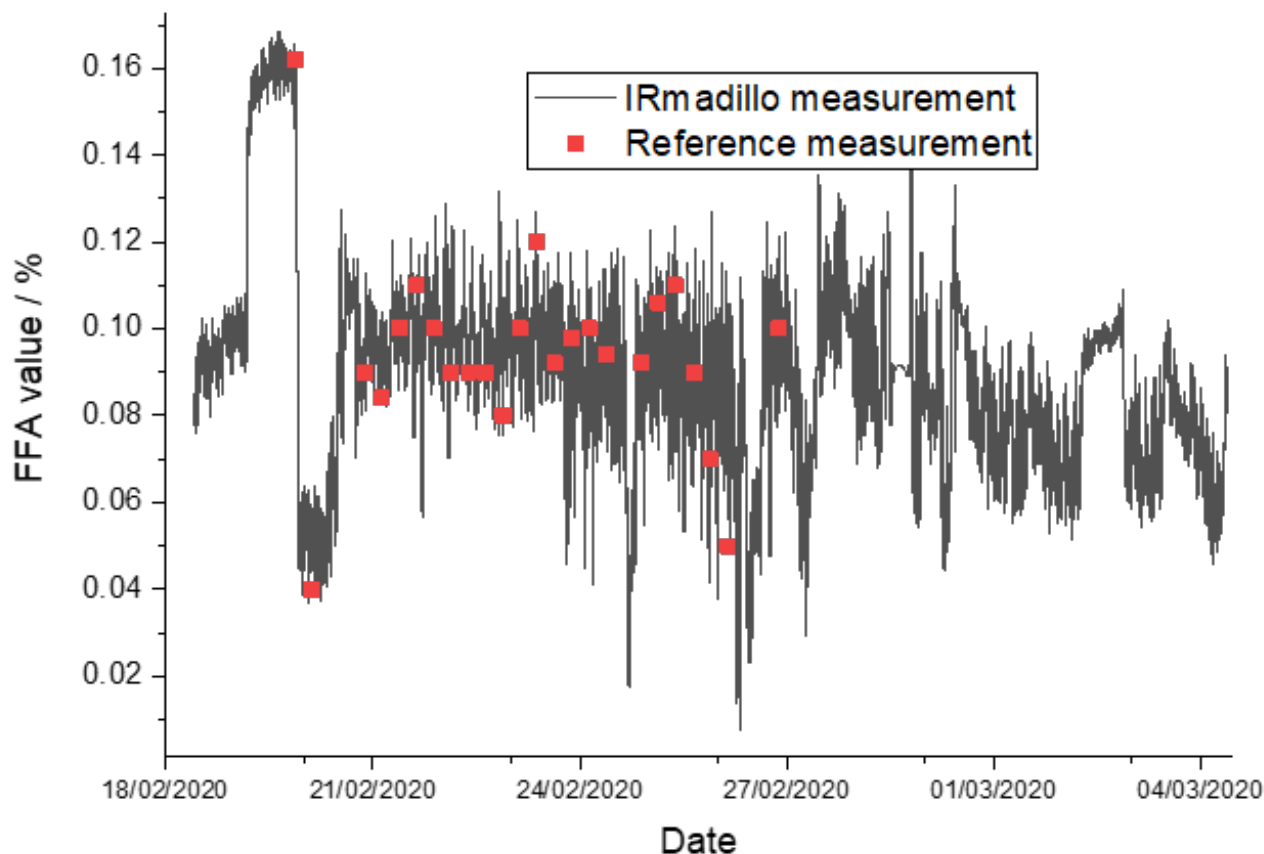
## Example use cases

This application note presents data collected at multiple sites in the UK and Europe. The instruments have been installed in either:

- Water washed oil inlet
- Bleached oil outlet

and have separate measurement calibrations built depending on the concentration ranges for FFA.

Once calibrated, the in-line/on-line and real-time measurements produced by the IRmadillo can be sent to the control room and DCS, enabling real-time control of caustic dosing or bleaching time, depending on the specific use case.



**Figure 1:** The measurement of FFA in bleached oil over several weeks using an IRmadillo analyzer in both rapeseed and sunflower oils

## Results

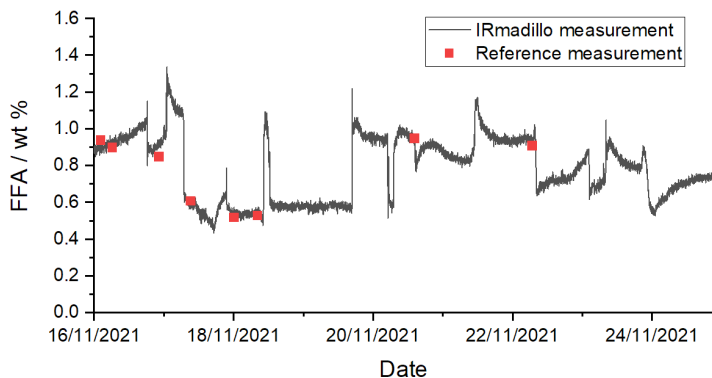
Global calibrations were made for all different types of FFA using PLS or SVR modelling (depending on installation type). Single calibrations can be built regardless of oil type - for example when process lines have both sunflower and rapeseed (or Canola) oils passing through them.

Typical sensitivities and measurement errors depends on exactly where the instrument is installed:

- Water washed oil inlet:  $\pm 0.2\%$
- Beached oil outlet:  $\pm 0.08\%$

Once implemented it is clear from the results that real-time measurement is highly valuable. Figure 1 shows the output of FFA measurement in bleached oil, and shows an upset in the process where the residual FFA raises from 0.09 to 0.16 % very quickly. This is not noticed by the refinery for 12 hr until a sample is taken by the laboratory, at which point the cost to rectify is high. The IRmadillo noticed the problem immediately.

The IRmadillo also works in water washed oil (and would also work in crude oil too), showing how the FFA value does change across a batch - see Figure 2 for the

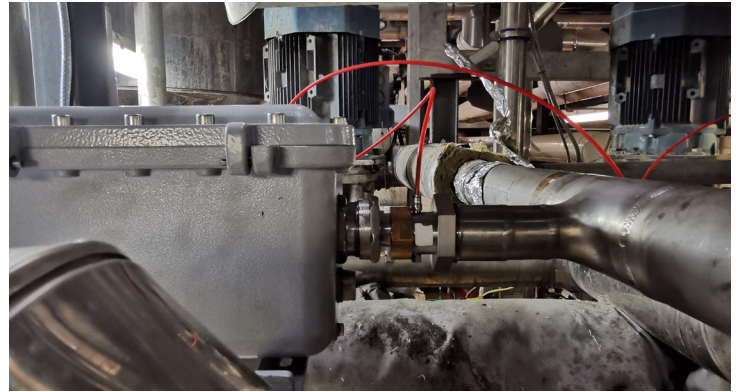


**Figure 2:** Measurement output of an IRmadillo installed in water washed oil inlet

## Keep in Mind

The IRmadillo can be calibrated to measure a very large range of chemicals, all in real time. It has effectively measured phosphorus (P), water/moisture and many different metals (Fe, Cu, Ca and Mg) simultaneously in a range of different oil types. It can even measure iodine value (IV), viscosity and melting point profiles. Once calibrated, the IRmadillo becomes a universal chemical concentration meter - for all types of fats and oil processing.

results. This has allowed the customer to better control their caustic addition - with an aim to implement real-time closed loop control for automated chemical addition.



**Photo:** IRmadillo instrument installed directly into a water washed oil inlet pipe using a simple T-piece

## Conclusions

This work has shown that the IRmadillo spectrometer is easy to install directly into various different locations in an oil refinery - whether at the inlet or outlet of processes.

The IRmadillo has been successfully calibrated to measure total FFA regardless of oil feedstock or the exact chemical species (i.e. palmitic, oleic or other types of acid) providing continuous and real-time measurements to the DCS. Additionally, the use of the instrument identifies problems in the process much earlier than manual off-line samples - as shown with the bleached oil example.



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