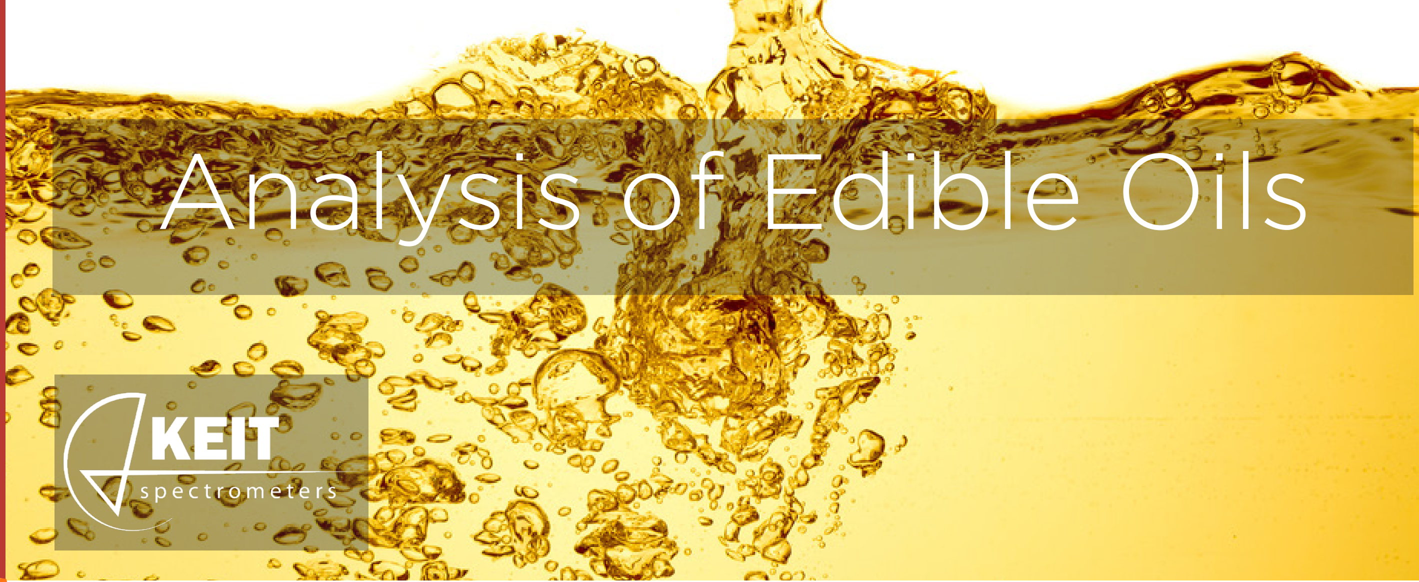
Analysis of Edible Oils



FTIR: The IRmadillo for Analysis of **Thermal Degradation in Edible Oils**

Introduction

Frying cycles within industrial operations involve oil being frequently reused and reheated at elevated temperatures. Unlike with home frying, the quality of these edible fats is significantly reduced, due to decomposition products forming in deteriorated oil.

The cycling at high temperatures combined with oxygen exposure and presence of water, degrade both the oil and food components, reducing acceptability and nutritive value of food.

At frying temperatures, major chemical reactions occur, and this reduces the stability of the frying oil, impacting safety, flavour and stability of fried food.

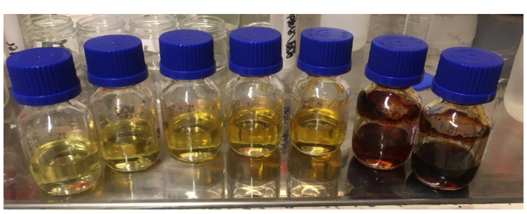
Challenges with Analysis

Most standard analytical methods for oil analysis are:

- Expensive
- Lengthy sample preparation times
- Depend on advanced instrumentation and user knowledge
- Experience model transfer issues and performance drift

Mid-Infrared Solution (FTIR)

The IRmadillo FTIR spectrometer offers a solution; as an easy to use, in situ instrument, it enables real-time analysis and quality control of industrial frying oils.



Visual example of thermal degradation of edible oil heated to 185°C.

Power of Mid-Infrared vs. NIR

Compared to Near Infrared (NIR), the IRmadllo FTIR (which is Mid-Infrared) with chemometrics can differentiate similar compounds.

- Mid-infrared is more precise and accurate than other wavelengths, and can be used to identify molecules and characterise their structure; NIR provides only the lower intensity overtone which is difficult to quantify and interpret
- Calibration data in Mid-IR is readily transferable from instrument to instrument compared to NIR
- NIR often experiences model and performance drift.

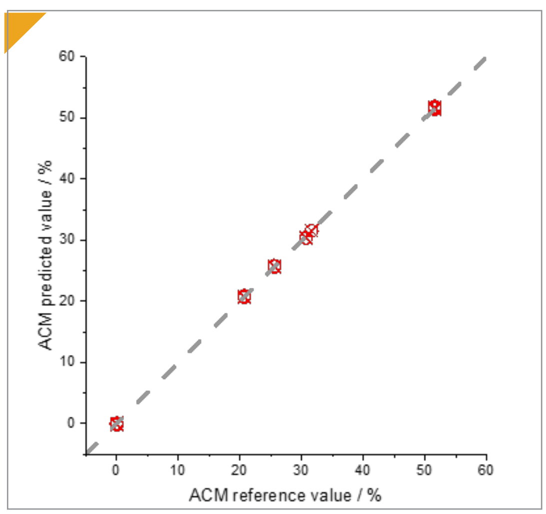


Figure 1: Additional Low-Level Analysis of Acrylamide.

Experimental

The IRmadillo analysed 8 samples of refined (flavourless, high smoke point) edible oil that had spent up to 192 hours in an oven at 185°C.

The system was sealed from ambient atmosphere under a N_2 purge, and a background scan was taken for 1 hour prior to sampling. After removal from oven, cold sample measurements were acquired for 120 s in batches of 3. The spectral range of 900 to 2000 cm^{-1} was used for analysis.

Sample #	Time in Oven/hrs	Sample #	Time in Oven/hrs
Sample 1	0	Sample 5	48
Sample 2	5	Sample 6	53.5
Sample 3	24	Sample 7	168
Sample 4	29	Sample 8	192

Spectra acquired during the experiment can be seen in Figure 2. IRmadillo FTIR spectroscopy of oxidative degradation of edible oil shows that the thermal stress of seed oils can be monitored via the amount of rising degradation product.

Figure 3 isolates a particularly key region of interest. Thermal oxidation exhibits a linear trend for increasing intensity at ~1700cm^{-1}, this observation corresponds to the formation of degradation products, in particular aldehydes (oxidation of Linoleic Acid). Secondly, the decreasing C=C stretching observed at ~1640cm^{-1} , illustrates chemical changes as a result of oxidation.

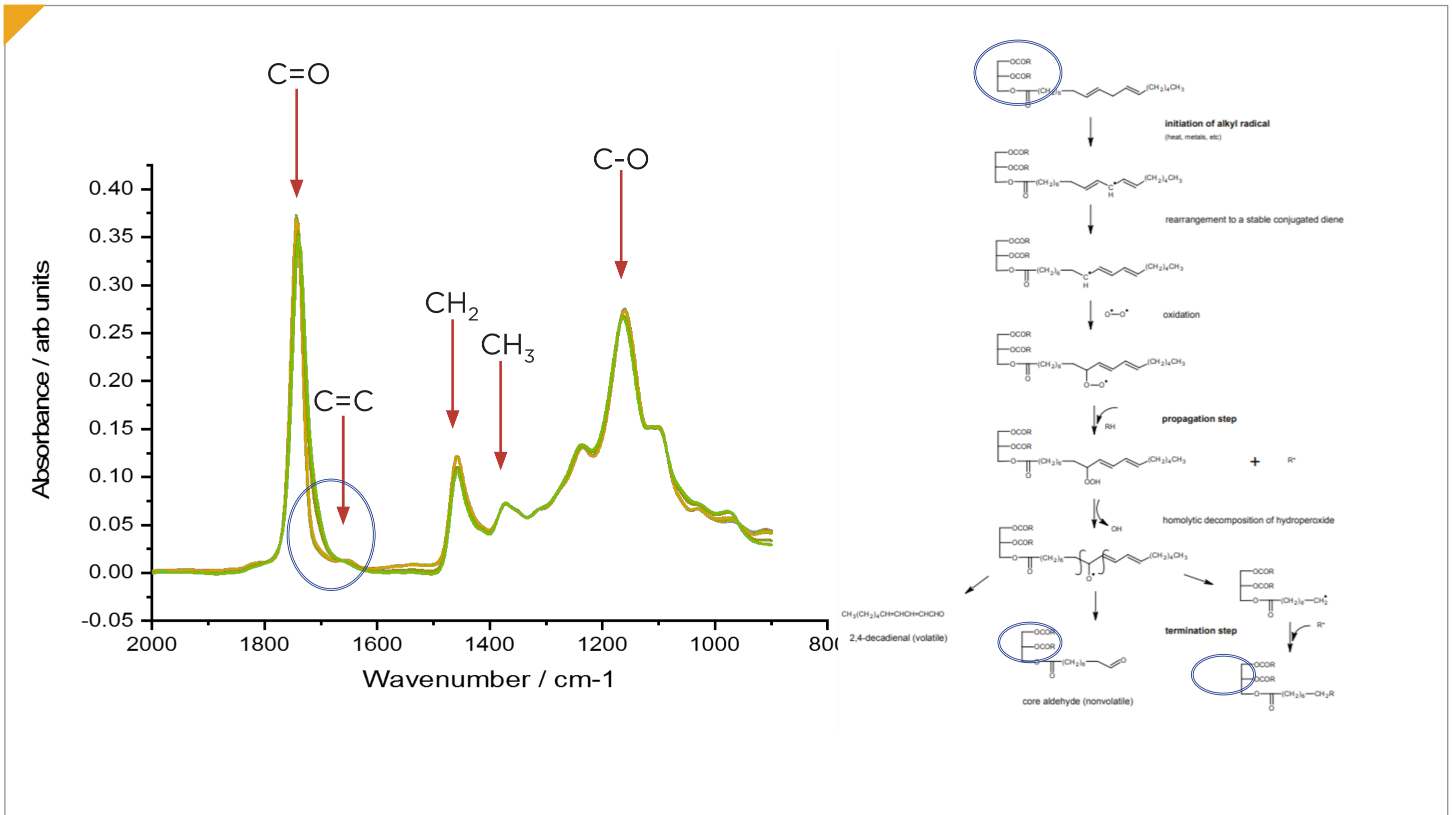


Figure 2: FTIR spectra of thermally degraded edible oil as recorded by the IRmadillo Spectrometer with 120 s sampling time between 900 to 2000 cm^{-1}. The arrows show key peaks and intensities.

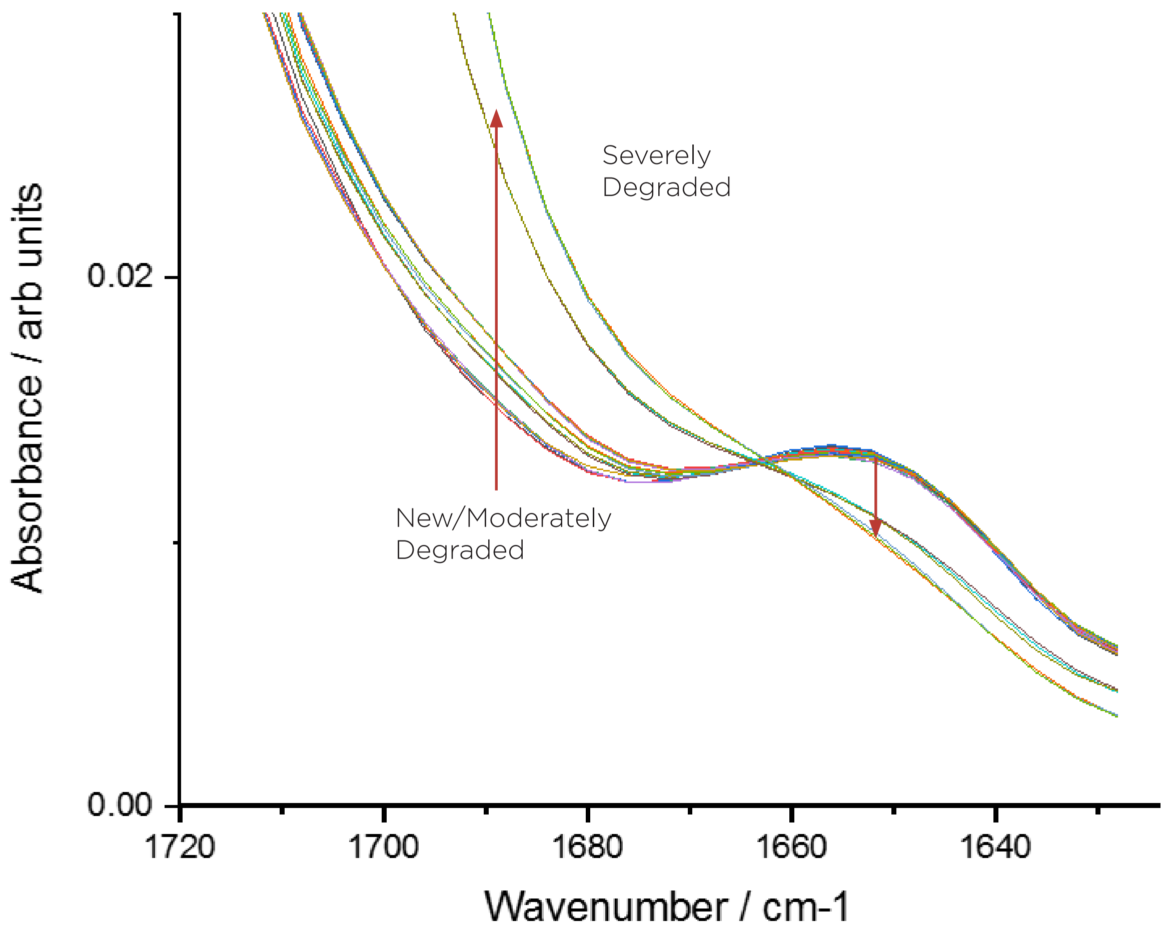


Figure 3: IRmadillo FTIR spectra of thermally degraded edible oil, isolating 1650 to 1720 cm^{-1} to demonstrate the differences in analytical spectra depending on the level of thermal degradation and oxidation.

Results and Discussion

The results from the chemometric model on the IRmadillo are shown in Figure 4. This plot shows the measured value from the IRmadillo on the y-axis vs the reference time value on the x-axis. A perfect correlation would be a straight line with a slope of 1. The black spots show the samples that were used to build the model, while the red spots show the results from samples that were not used in the model—these are true predictions.

It is immediately apparent from the graphs that there is excellent correlation between the measured and reference samples. This means that the IRmadillo can accurately measure oil degradation with time. The data was analysed using Extended Multiplicative Scattering (EMSC) and Partial Least Squares (PLS) models to predict. The IRmadillo can detect thermal degradation within 3 hours. Utilising PLS, allowed the data structure in terms of sample patterns to be analysed (scores); each oil sample demonstrated differences with time, and linearity was observed as the spectra features associated with thermal degradation became a more prevalent in describing the oil samples.

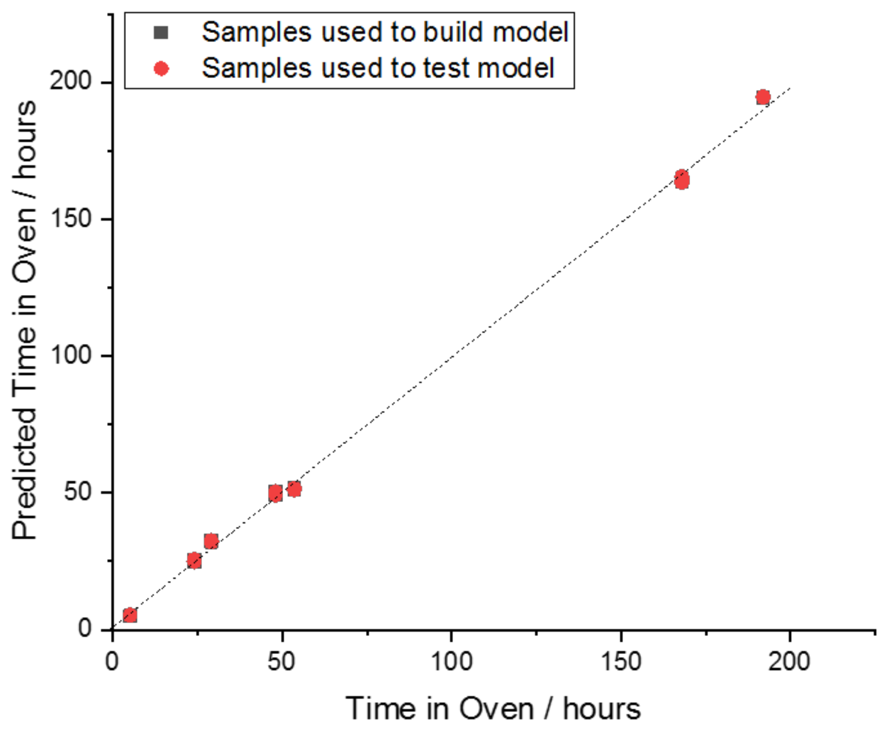


Figure 4: Plot of predicted vs actual time values for edible oil thermal degradation in oven.

Conclusions

Oxidation is the main reaction in the degradation of lipids. With the risk of reduced acceptability and nutritive value of fried food, it is essential to know the composition of fatty acids in edible oil.

Traditional methods of analysis (HPLC, GC, UV-Vis) do not provide the possibility to couple with any automatic control element for process control. NIR has its own limitations on spectral range, model transferability and spectral drift.

FTIR spectroscopy is a rapid and reliable technique that could precisely determine edible oil stability during processing and storage without the use of reagents and solvents.

Real-time analysis with the IRmadillo FTIR spectrometer combined with chemometrics has the potential to provide a rapid screening method to monitor lipid quality and process control, as it can successfully characterise thermal degradation of edible oils.

Keep in mind

There may also be other compounds present during a process which may be of interest to you.

As an FTIR spectrometer, the IRmadillo observes not just a single element, but all the compounds of a mixture. It can track multiple compounds simultaneously to provide a full picture of your process, with monitoring tailored to your needs.

IRmadillo™ FTIR Spectrometer

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