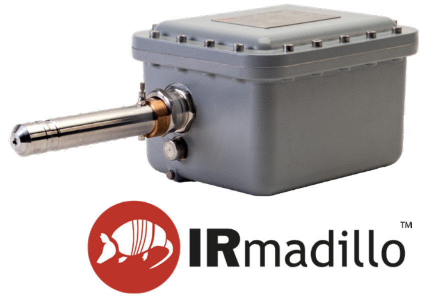
Analysis of Edible Oils

Detection of Adulteration in Edible Oils
with the IRmadillo On-Line Process Analyser



Abstract

The IRmadillo FTIR spectrometer was used to determine authenticity and adulteration of Extra Virgin Olive Oil (EVOO) with Soybean Oil (SO). The IRmadillo could detect adulterated Soybean Oil in EVOO as low as 1.4%. It could also differentiate when EVOO was adultered with Rapeseed Oil (RO) (LoD 2.5%), and identify trends in levels of adulteration between with different seed oils.

Introduction

Cooking oil, particularly Olive Oil, can be adulterated with less expensive seed oil. EVOO contains the most health benefits due to its high nutritional value. EVOO can be adulterated with refined oils or less expensive oils, such as Rapeseed Oil (Canola Oil) and Soybean Oil, for financial gain.

It is one of the most commonly adulterated food products, due to its low production and higher prices. Oils are composed mainly of triacylglycerols (derived from the esterification of three fatty acid molecules with a glycerol molecule). All oils contain a different composition of fatty acids, this means that when the IRmadillo FTIR (mid-infrared) spectrometer is combined with chemometrics, these Olive Oil adulterants can be differentiated.

Challenges with Analysis

Most standard analytical methods (such as HPLC, GC, UV-vis and NIR) 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, on-line instrument, it enables real-time analysis and quality control of industrial edible oils.

Advantages of FTIR vs. NIR

Compared to Near Infrared (NIR), the Fourier Transformed Infrared (FTIR) IRmadllo (which is Mid-Infrared) has distinct advantages:

- FTIR (Mid-IR) 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 the mid-IR is readily transferable from instrument to instrument compared to NIR
- NIR often experiences model and performance drift.

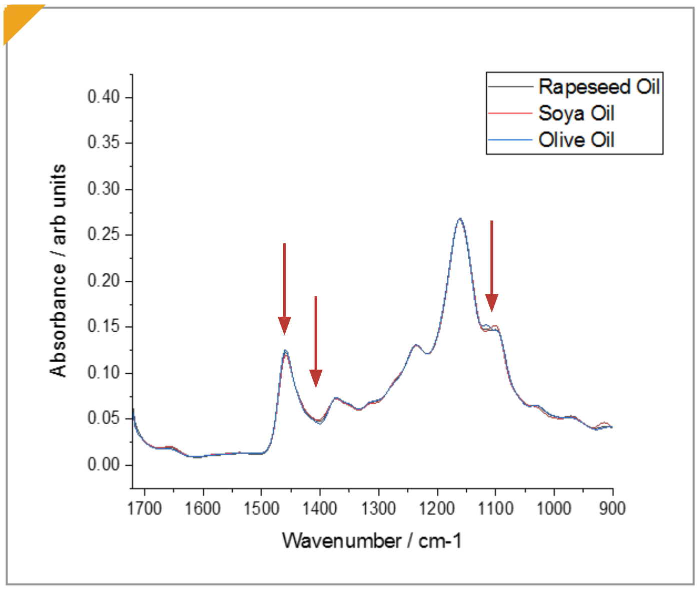


Figure 1: FTIR spectra of Rapeseed Oil, Soybean (Soya) Oil and Olive Oil as recorded by the IRmadillo Spectrometer with 120 s sampling time between 900 to 1740 cm^{-1}. Key differentiating features are highlighted.

Experimental

The IRmadillo was initially used to analyse adultered EVOO with Soybean Oil in ratios of 100/0, 80/20, 85/15, 90/10, 91.5/8.5, 93.5/6.5, 95/5, 0/100.

The system was sealed from ambient atmosphere under a N_2 purge, and for each, a background scan was taken for 1 hour prior to sampling. Sample measurements were acquired for 120 s in batches of 3. The spectral range of 900 to 1470 cm^{-1} was used for analysis. The Limit of Detection (LoD) for Soybean Oil in EVOO is 1.4%.

The results from an Extended Multiplicative Scattering Correction (EMSC) and Partial Least Squares (PLS) model demonstrates good agreement, and can be seen in Figure 3. A small number of adultered EVOO with Rapeseed Oil samples were also analysed to compare.

Spectra acquired during the experiment can be seen in Figure 1. The IRmadillo can clearly differentiate between Rapeseed Oil, Soybean Oil and Olive Oil. Following this, EVOO was adultered with 5 and 10% Rapeseed Oil, a 50% mix of Rapeseed Oil and Soybean Oil, and equal mix of all Oils was prepared.

When all the results were analysed following EMSC and PLS models, the scores plot results illustrated distinctions between all groups. These distinctions are made by determining the variables that maximise the variation between groups and those which minimise variation within groups. The score describes the data structure in terms of sample patterns and highlight sample similarities and differences.

Authentic EVOO was clearly separated from the adulterant groups and the seed oils. As the level of adulteration increased, a pattern of linearity was observed as the variables describing the data shifted away from EVOO and towards either RO or SO. The score for the 50% mix was positioned between the two authentic seed oil clusters, and the equal mix of oils was centred between all the authentic oil clusters. The LoD for Rapeseed Oil in EVOO is 2.5%.

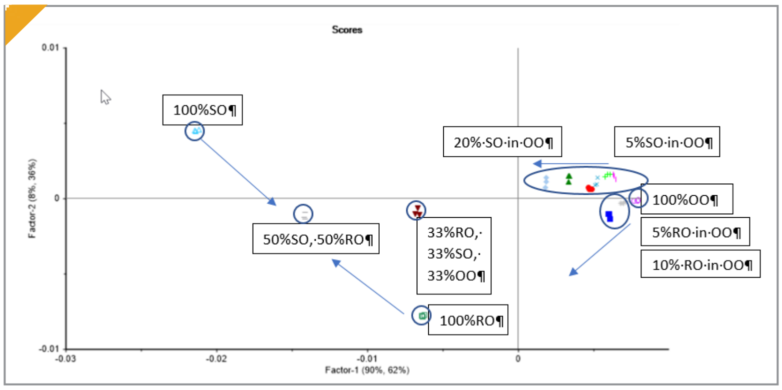


Figure 2: Combined with chemometrics, IRmadillo FTIR spectrometer shows differences between oil blends.

Conclusions

Combing the IRmadillo spectrometer with chemometrics has shown to be effective in identifying EVOO as being distinct (due to EVOO containing more oleic acid and less linoleic and linolenic acids than the seed oils) from different types of seed oil, and when adulterated. This system is also effective in differentiating between different types of seed oils, and can be utilised in analysis of contaminants, impurities and adulteration.

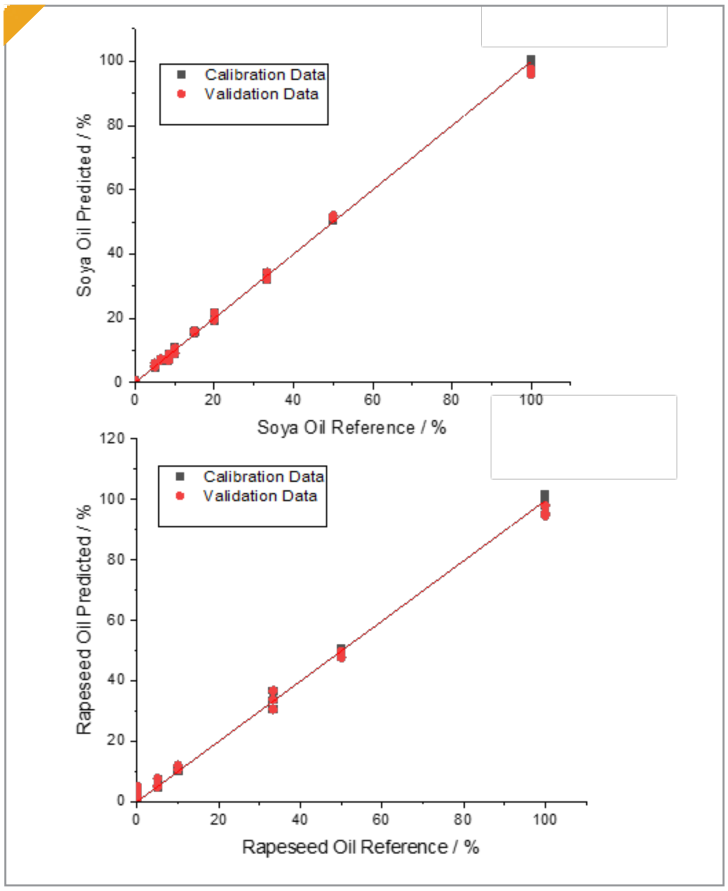


Figure 3: Spectra of Rapeseed Oil, Soybean (Soya) Oil and Olive Oil as recorded by the IRmadillo Spectrometer with 120 s sampling time between 900 to 1470 cm^{-1}. Key differentiating features are highlighted.

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