



Quality control of Virgin Olive Oil by Near-Infrared Spectroscopy



Lagardère L., Lechat H., Lacoste F.

ITERG - Institut des corps gras - f.lacoste@iterg.com

A rapid Fourier transform near-infrared (FT-NIR) spectroscopic method was developed for quantitative determination of peroxide value (PV) and oleic acidity (OA) of virgin olive oils. Calibration models were constructed using Partial Least Squares (PLS) regression with different wavenumber ranges using about 60 samples. The best results were obtained between 5100 and 4520 cm^{-1} for oleic acidity and between 7500 and 6300 cm^{-1} for peroxide value. Models were verified with a set of 15 samples. FT-NIR allows the prediction of PV with an uncertainty of 1.0 meqO_2/kg within a 3 to 32 meqO_2/kg analytical range, and the prediction of OA with an uncertainty of 0.06% within a 0.15 to 1.3% analytical range. Due to its simplicity and rapidity, FT-NIR method provides an alternative mean for determination of quality parameters of virgin olive oils.

Introduction

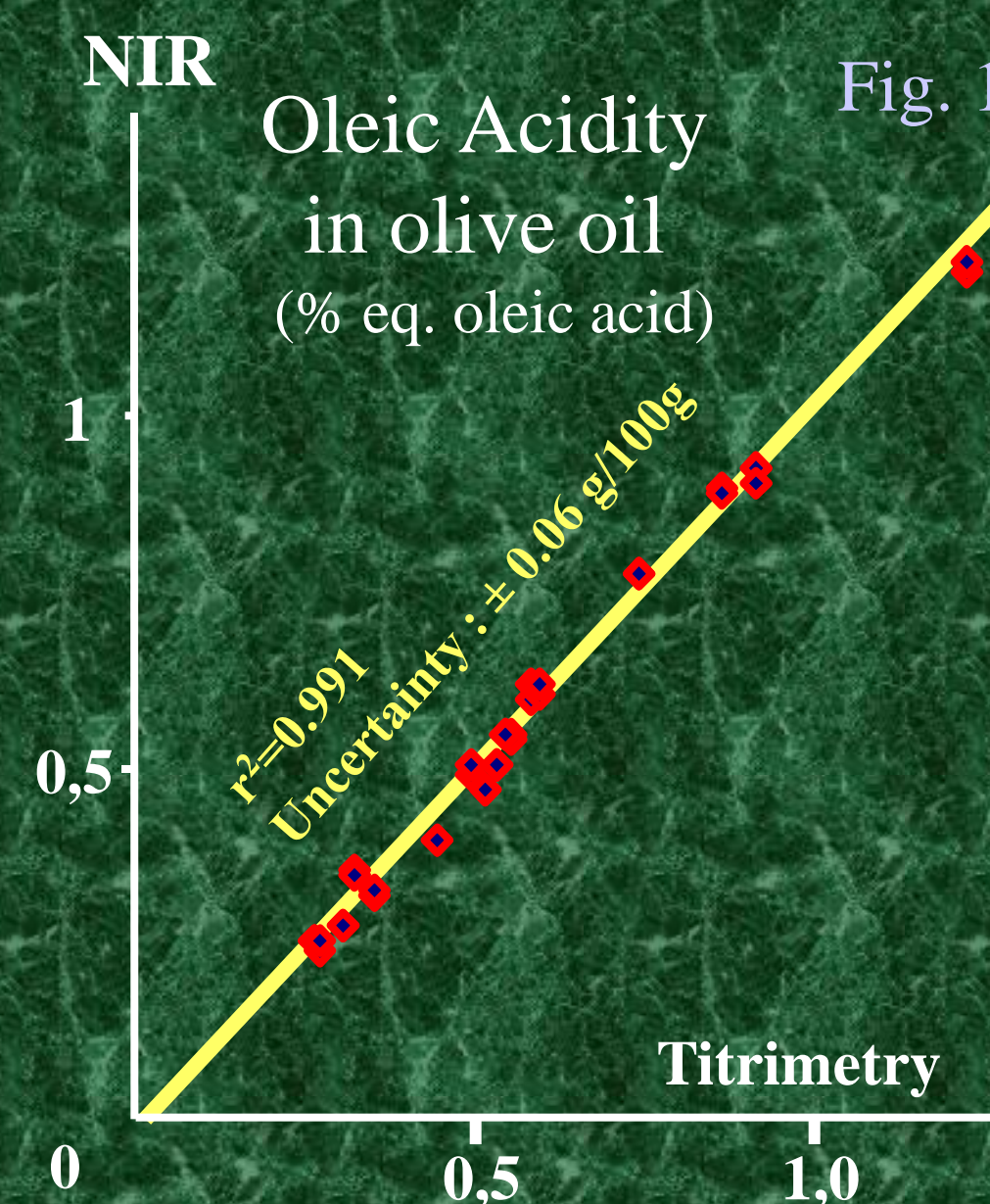
Peroxide Value (PV) and oleic acidity (OA) are amongst of the best indicators of oil degradation status. These values are usually determined by volumetric titration, which is destructive, time consuming and requires important volume of chemical products. Since a few years, Near Infrared (NIR) Spectroscopy revealed to be an interesting alternative technique for the analysis of foods and especially of edible oil. Indeed, an alternative method for the determination of Iodine Value was developed and standardized as AOCS Cd 1e-01. The objective of this work was to evaluate the quality of determination of IP and OA by the NIR technique on olive oil.

Material and methods

A Bomen model MB 160 NIR (ABB Bomem, Quebec, Canada) Fourier-transformed Near Infrared Spectrometer (FT-NIR) was used for all the spectrometric analysis. A spectrum from 12000 to 4000 cm^{-1} was measured and calculation was realized by Partial Least Square (PLS) regression using Bomem Grams software.

60 commercial extra virgin olive oils samples from different origins, obtained from retailers, were used for calibration and 15 others were reserved for validation. Reference PV and OA were determined in duplicate on every oil using NF T 60-220 (equivalent to AOCS Cd 8-53) and EN ISO 660, respectively

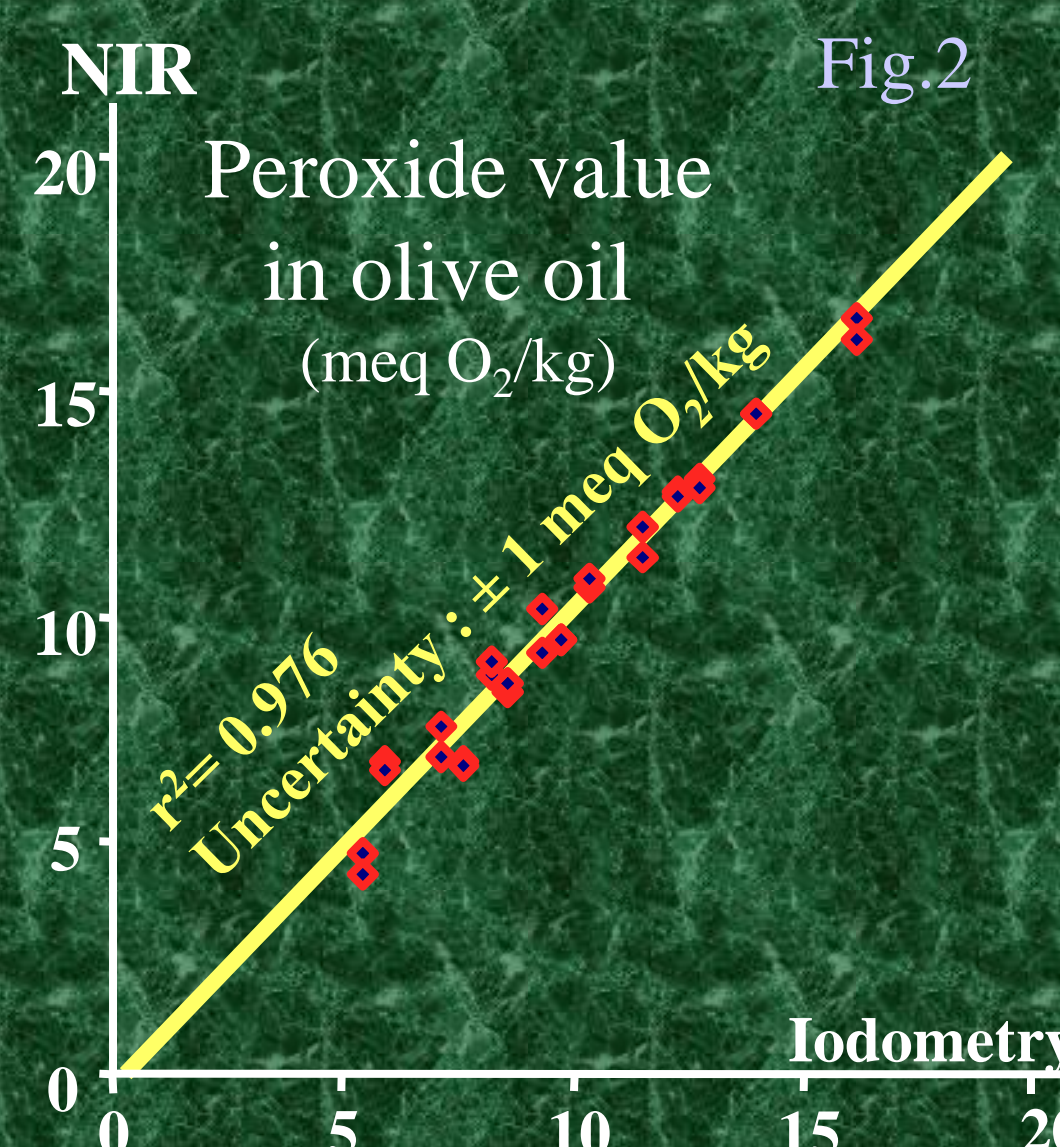
Measure of oleic acidity with FT-NIR



The specific signal of the acidity is very intense in the 5100 to 4520 cm^{-1} region. This region, corresponding to C=O elongation, allowed very satisfactory calibration model ($r^2 > 0.99$) which was confirmed with the validation samples (Fig. 1).

Measure of peroxide value with FT-NIR

Specific IR absorption associated with peroxide content showed to be less intense than free acidity. The maximum difference was observed in the spectral region 7500 to 6350 cm^{-1} , which correspond to -OO-H elongation. However, Specific IR absorption associated with

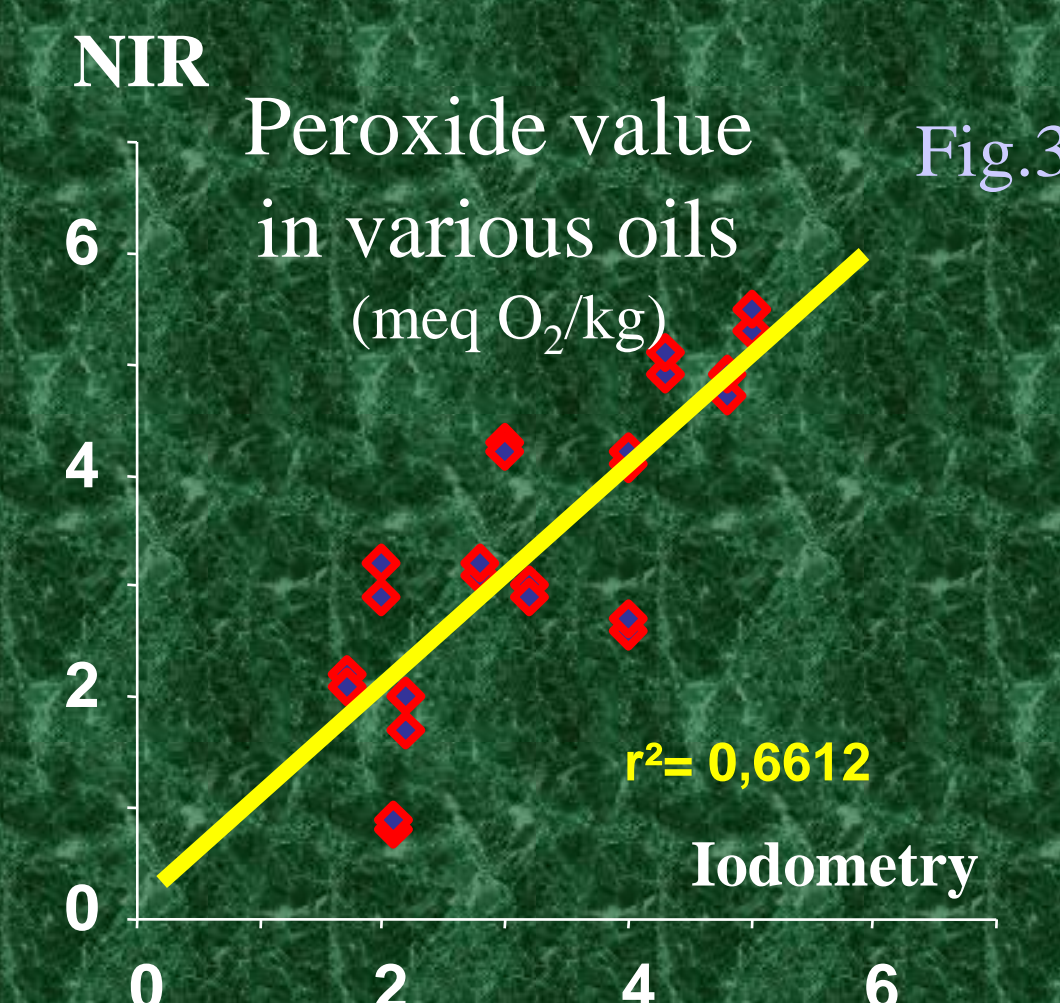


peroxide content was sufficient to give good correlation between titration and FT-NIR methods (Fig.2). The uncertainty measured on validation samples was very similar to the uncertainty associated with standard method, and is enough for the olive oil quality control.

Measure of PV and OA on various type of oils

Calibrations tested on various type of oils were not conclusive : correlation was too low to allow good prediction of these values (fig.3).

But, working on sunflower oil allowed to get satisfying calibration.



Conclusion

Analysis of peroxide value and acidity by FT-NIR analysis is a good alternative to standard titrimetric methods. Peroxide value measurement can be made with a 1 $\text{meq O}_2/\text{kg}$ uncertainty and oleic acidity with a 0.06 % uncertainty, which is almost as good as the standard method. However, this method is oil specific as a specific calibration model is needed for each oil.