

# A Performance Comparison of Two Portable NIRS Technologies for Olive Oil Adulteration

Ana Soladado

*Dept. of Physical and Analytical  
Chemistry  
University of Oviedo  
Oviedo, Spain  
soldadoana@uniovi.es*

Jose Manuel Costa

*Dept. of Physical and Analytical  
Chemistry  
University of Oviedo  
Oviedo, Spain  
jcostafe@uniovi.es*

Candela Melendreras

*Dept. of Physical and Analytical  
Chemistry  
University of Oviedo  
Oviedo, Spain  
melendrerascandela@uniovi.es*

Patricia Lozano Fernández

*Dept. of Physical and Analytical  
Chemistry  
University of Oviedo  
Oviedo, Spain  
UO252766@uniovi.es*

Juan Carlos Campo

*Dept. of Electrical, Electronic,  
Communications and Systems  
Engineering  
University of Oviedo, Gijón, Spain  
campo@uniovi.es*

Marta Valledor

*Dept. of Electrical, Electronic,  
Communication and Systems  
Engineering  
University of Oviedo, Gijón, Spain  
valledormarta@uniovi.es*

Alberto López

*Dept. of Electrical, Electronic,  
Communications and Systems  
Engineering  
University of Oviedo, Gijón, Spain  
albertolpezm@gmail.com*

Francisco Ferrero

*Dept. of Electrical, Electronic,  
Communications and Systems  
Engineering  
University of Oviedo, Gijón, Spain  
ferrero@uniovi.es*

**Abstract**—To date, the development of low-cost, easy to use real time and no-invasive sensors for safety and quality controls in agrifood is needed. One of the most promising techniques that complies with these requirements is Near Infrared spectroscopy (NIRS). In this work, two portable NIRS (Nanoscan Texas Instruments, and MicroNIR 1700, Viavi). have been tested as a feasible sensor to detect frauds in olive oils (olive oils adulterated with low-cost seed oils). Spectra of 99 oils were scanned in transmittance (Nanoscan) and reflectance (MicroNIR) modes. After transforming raw data with derivative and scattering math pretreatments, Partial Least Square regression models were developed. Results of Principal Component analysis (PCA) with Nanoscan raw data showed that exist differences between Olive oil and adulterated oils. In both portable NIRS devices, the coefficients of determination for calibration for all adulterations were at least 0.8.

**Keywords**—Oil adulteration, spectrometer, near-infrared (NIR), principal component analysis.

## I. INTRODUCTION

Near-infrared (NIR) spectroscopy (NIRS) is important tool for measuring the properties of material through the variation of light absorbed by the sample in the range from 750 to 2500 nm. Spectroscopy is used in many areas of application such as pharmaceuticals, medical, food, agriculture, etc. because of its high performance and affordable spectrometers [1, 2].

It is of significant value to the food industry the development of NIRS-based methodologies that identify food frauds in the agri-food chain, from producers to inspection services. Lab methodologies require expensive instrumentation and expert operators. Traditional methods, such as high-performance liquid chromatography (HPLC) or gas chromatography [3,4], are not able to measure the quality of certain oil manufacturing processes.

A non-invasive alternative procedure to analyse the quality and safety parameters of oil in real-time is the NIRS-based procedures. However, developing and implementing a

NIRS-based procedure at the industry or field level is not easy. Miniaturized NIRS instrument technical characteristics, sample presentation and scan collection strategies are critical to obtaining a valuable spectrum of intact samples and developing a chemometric model to assess food quality and safety [5]. [6] is a special issue about the current state-of-the-art of NIRS, for predicting quality compounds in foods.

It is well established that NIRS is a real-time and non-destructive technique that can be used for quality and safety controls in olive oil. The speed up on providing olive oil quality results make NIRS an alternative to be proposed as an official method to determine quality control parameters. However, the usefulness of the proposed methodology depends on the quality of NIRS spectra. Spectra collected with high performance instruments and adequate sample cups provide high quality NIS data and robust calibration models. However, this type of instrumentation is expensive and is not available to operators in the agricultural sector. These reasons have done necessary to seek low cost NIRS and simple sampling strategies able to provide spectra that can be used to develop NIR robust calibrations. In this work two different instruments a homemade prototype (NIRscan nano) and a commercial one (MicroNIR), and two different scanning modes (transmittance and Reflectance) and cuvettes have been evaluated in order to select the best option to establish quality control in olive oil to quantify adulteration with other oils.

In recent years portable NIR spectrometers have improved their performance considerably. Different technologies are nowadays available, such as FT-NIR (Si-Ware), linear variable filter (Viavi MicroNIR), and diffraction grating (Texas Instruments NIRscan Nano). In [7] Pasquini analyses the advances in the last decade showing the innovative aspects of NIRS. A very complete review (582 references) of portable spectroscopy and its applications was provided by Crocombe [8]. A comprehensive overview of miniaturized NIR spectrometers is provided by Béc et al. [9], including the

technology's principles, current applications, and potential advances in the future.

Several studies have been conducted on olive oil adulteration detection [10–14]. To compare the proposed technique in this work (portable NIR) with the most widely used techniques, Table I shows the pros and cons of each technology.

TABLE I. MAIN TECHNOLOGIES USED FOR DETECTING OLIVE OIL ADULTERATION

Technique	Advantages and Disadvantages
Portable NIR (This work)	<ul style="list-style-type: none"> <li>• Samples are not pretreatment</li> <li>• Analyses in real-time and on-site</li> <li>• Inexpensive lab instruments</li> <li>• Large database for model calibration</li> </ul>
Fluorescence Spectroscopy [15]	<ul style="list-style-type: none"> <li>• Samples are not pretreated</li> <li>• Expensive lab instrumentation</li> <li>• Each adulteration type has its own model</li> </ul>
Medium Infrared Spectroscopy [16]	<ul style="list-style-type: none"> <li>• Easy and rapid implementation</li> <li>• Expensive lab instrumentation</li> <li>• Mathematical equations can be unstable</li> </ul>
UV – VIS and VIS – NIR [17]	<ul style="list-style-type: none"> <li>• Fast analysis</li> <li>• Implementable in the lab easily</li> <li>• Expensive lab instruments</li> </ul>
Near Infrared Spectroscopy [18]	<ul style="list-style-type: none"> <li>• Samples are not pretreated.</li> <li>• Non-damage analysis</li> <li>• Time-consuming calibration process</li> <li>• Costly lab instrumentation</li> </ul>
Raman Spectrometry [19]	<ul style="list-style-type: none"> <li>• Non-damage analysis</li> <li>• Preparation of a minimum sample</li> <li>• Fast approach</li> <li>• Samples' fluorescence produces interferences</li> <li>• Costly lab instrumentation</li> </ul>
E-nose Voltammetric [20]	<ul style="list-style-type: none"> <li>• High accuracy, reproducibility, and sensitivity</li> <li>• Costly lab instruments</li> <li>• Necessary specialist personal</li> </ul>
Gas Chromatography [21]	<ul style="list-style-type: none"> <li>• Conventional method</li> <li>• Accuracy, precision, and reproducibility</li> <li>• Pretreatments consume much time</li> <li>• Costly lab instruments</li> </ul>
High-Performance Liquid Chromatography [22]	<ul style="list-style-type: none"> <li>• Accurate, precise, and reproducibility</li> <li>• Sample pretreatments consume much time</li> <li>• Expertise personnel required</li> </ul>
Nuclear Magnetic Resonance [23]	<ul style="list-style-type: none"> <li>• Quick analysis time</li> <li>• Simple preparation of the sample</li> <li>• Excellent reproducibility but low sensitivity</li> <li>• Costly lab instrumentation</li> <li>• Required specialist personal</li> </ul>

This work was focused on the following contributions:

- 1) A comparative study of the most used analytical techniques for detecting olive oil adulteration.
- 2) Evaluation of two portable NIR spectrometers and two scanning modes, transmittance and reflectance, for olive oil adulteration.

The rest of the article is structured as follows: Section II outline the main features of two NIRS technologies that can be used for olive oil adulteration. In section III, MicroNIR and NIRscan nano spectrometers were used to scan oil samples. Criteria for selecting an olive oil adulteration instrument can be found in Section IV. To conclude, Section V includes the conclusions.

## II. PORTABLE NIRS TECHNOLOGIES

This section compares two portable micro-spectrometers based on different portable spectrometers using different technologies: NIRscan Nano evaluation module (EVM) manufactured by Texas Instruments Inc. (TX, USA) and the MicroNIR 1700 manufactured by Viavi Solutions Inc. (CA, USA). These instruments will be used in section III for evaluation of olive oil adulteration. Table II shows the main specifications of both micro-spectrometers. These features have been taken from the manufactures and will be discussed in the following two subsections.

TABLE II. SPECIFICATIONS OF NIRSCAN NANO AND MICRONIR 1700

Key features	NIRscan Nano EVM	MicroNIR 1700
Wavelength Range	900–1700 nm	950 –1650 nm
Illumination source	Two tungsten halogen lamps	Two tungsten halogen lamps
Wavelength selector	Grating and MEMS DMD	Linear Variable Filter (LVF)
Spectral Resolution	10–12 nm	6.2 nm
Detector	InGaAs (single element)	128-pixel InGaAs photodiode array
Analog-to-digital convertor	24 bits	16 bits
Signal-to-noise ratio (typical)	6,000:1 (Reflective head)	23,000:1
Scan rate or Integration time	0.3 s	0.25–0.50 s
Weight	85 g	64 g
Cost (VAT included)	\$1250 [24]	\$14500

### A. Texas Instruments NIRscan Nano Spectrometer

Fig. 1 illustrates a simplified view of the optical path of the NIRscan Nano Evaluation Module (EVM) from Texas Instrument [25]. It includes a sapphire window, two low-end tungsten filament lamps, collection lens, collimating and focusing lenses, a reflective diffraction grating, a digital micromirror device (DMD) and a single light detector (InGaAs) [26]. In a DMD, there are thousands of very small micromirrors. By turning on or off mirror columns, individual wavelengths can be selected. This is done by reflecting only the desired wavelengths to the detector. DLP DMD's architecture allows the use of a large, single light detector instead of an expensive array light detector in a spectrometer.

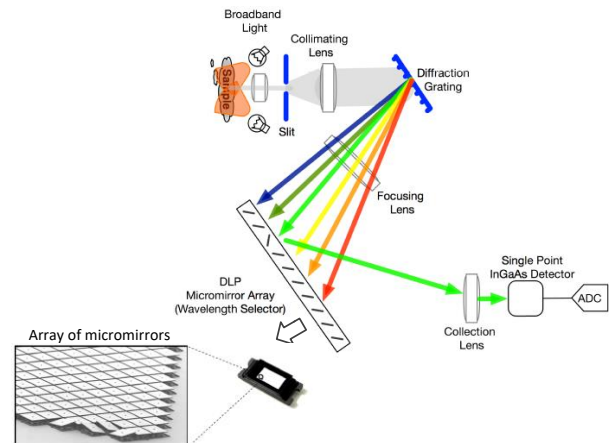


Fig. 1. DLP-based spectrometer from Texas Instruments.



Fig. 2. Texas Instruments DLP NIRscan Nano dimensions.

Fig. 2 shows an image of the NIRscan Nano EVM and its dimensions. This spectrophotometer has a unique feature: users can download the software and hardware documentation from the manufacturer's website. This is especially useful for developing specific applications for the instrument [27].

An important feature is the embedded Hadamard multiplexing approach to improve the signal-to-noise ratio of the measurements. This technique allows to observe more than one wavelength at a time using a mask. To achieve a programmable Hadamard mask, the micromirror array columns can be separately accessed. In a Hadamard matrix each element can be zero or one depending on if the element is included or not in the multiplexed measurement. Multiplexed measurements are the same as sequential measurements obtained by normal scans. As a result, a multiplexed measurement requires the same time it takes to scan a spectrum sequentially.

### B. VIAVI MicroNIR 1700 Spectrometer

VIAVI MicroNIR is based on a different approach [28]. The two main difference with respect to the TI NIRscan are an more expensive array detector (InGaAs) and a Viavi linear variable filter (LVF). This spectrometer is powered from USB. This spectrometer supports three main light modes: reflective, transmissive and transfective.

The very small size of the MicroNIR (50 x 45 mm) is based on a linearly variable filter (LVF) used as the diffraction element. Fig. 3 illustrates the working principle of this element. It consists of a dielectric thin-film Fabry-Perot bandpass filter deposited using energetic processes. The coating thickness modulates the wavelength of the bandpass filter therefore the peak of the wavelength transmitted vary with the wedge direction.

The LVF is directly attached to the linear detector array and coupled to the readout electronics and the diffuse tungsten illumination system. Figs. 4a and b show the VIAVI MicroNIR 1700 spectrometer and the optical design operating in diffuse reflection mode, respectively.

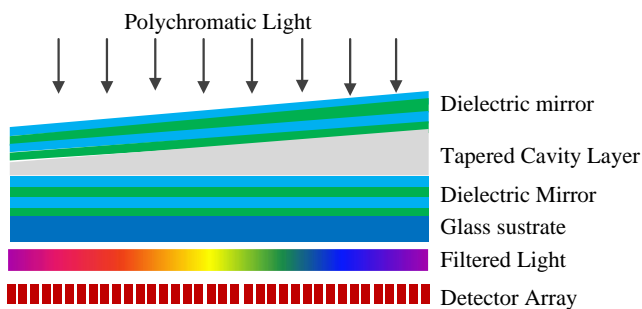
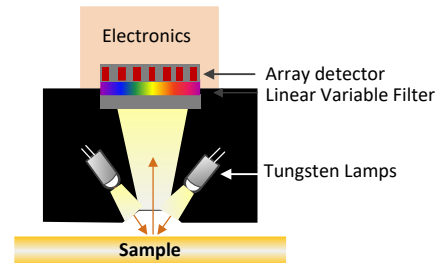


Fig. 3. Schematic of a linearly variable optical filter.



a)



b)

Fig. 4. a) The VIAVI MicroNIR 1700 spectrometer, b) MicroNIR optical design in diffuse reflection mode.

### III. MATERIALS AND METHOD

A database based on olive oil was created using the following types: extra virgin olive oil (EVOO), virgin olive oil (VOO), and olive oil (OO). These olive oils were adulterated with flax (FO), sunflower (SO) and sesame (SEO) oil. The adulteration percentage ranged between 2 and 30% for all evaluated olive oils. Fig. 5 shows a scheme of adulteration samples included in the sample population.

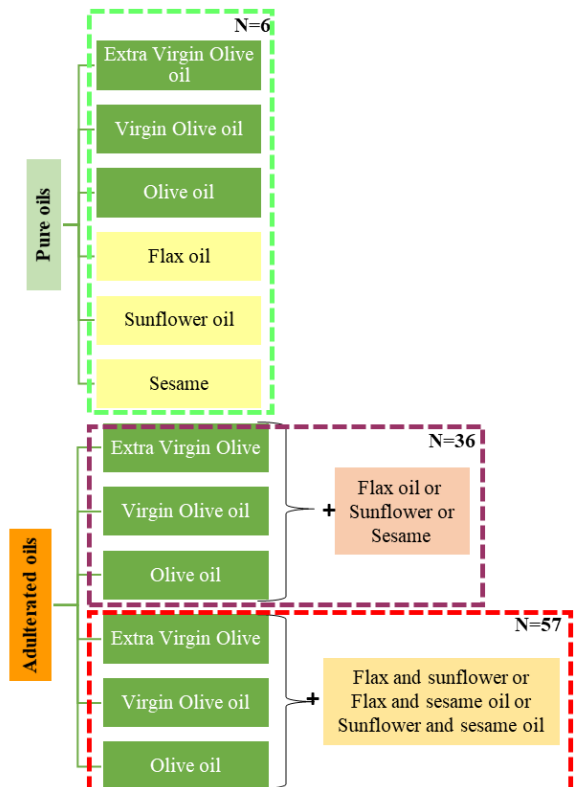


Fig. 5. Scheme of pure and adulterated oils (N=99) employed to build up spectra database.

Both MicroNIR and NIRscan nano were used to scan oil samples without prior treatment. MicroNIR spectra were collected in reflectance mode, using 5 ml glass vials. A quartz cuvette with an optical path of 10×10 mm was used for NIRscan nano measurements in transmittance mode. Instrumental conditions were as follows: for MicroNIR, spectra were collected using an integration time of 0.1 ms and averaging 100 scans per sub-sample; and for NIRscan nano, spectra were collected in Column mode, averaging 30 scans per sub-sample. All samples were scanned in duplicate mode (two sub-samples per sample) and the final spectra were the average of two spectra. The total number of data points defining each spectrum was 228 and 125 for NIRscan nano and MicroNIR respectively.

By combining spectra data and oil composition, matrix data was built for each NIR instrument. Unscrambler X software (CAMO Analytics AS, Oslo, Norway) was the chemometric software used to evaluate and compare both NIR instruments and design a strategy to identify olive oil frauds. Principal Component Analysis and Partial Least Square approaches were used to develop qualitative and quantitative strategies and chemometric models to detect olive oil fraud. The use of derivatives, e.g., Savitzky-Golay's (SG) first or second derivative or scatter correction procedures (such as standard normal variation SNV) can be useful when working with NIR data.

#### IV. RESULTS AND DISCUSSION

It is well established that successful results of developed models depend on the quality of the collected spectra. Fig. 6 shows the raw spectra of pure oils involved in this study and collected in both instruments. As can be seen in Fig. 6 some differences were observed using both instruments.

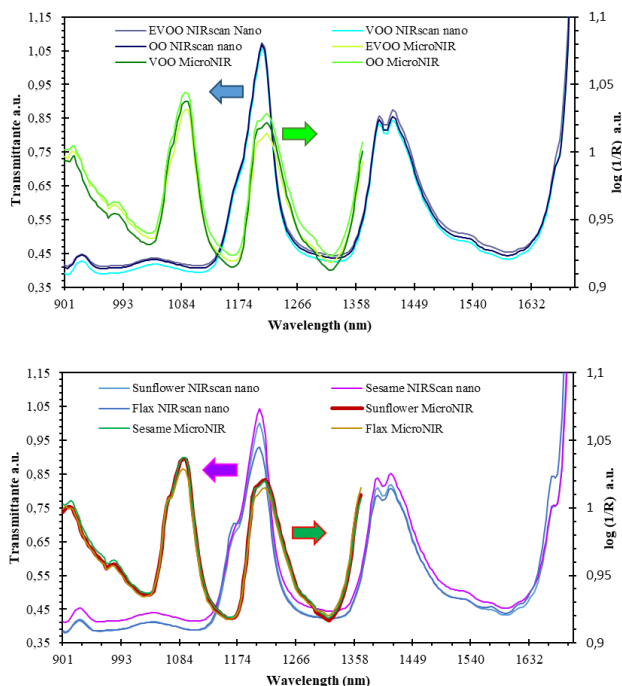


Fig. 6. Near Infrared Spectra of Pure oils using both instruments Micro and Nano. (R: Reflectance).

First, the band around 1084 nm in reflectance mode (MicroNIR) is not detected in transmittance mode (NIRscan nano). These spectra characteristics are quite similar to those obtained by García-Martín [29]. It is worth noting that the band between 1350-1450 nm (NIRscan nano) has been utilized by previous authors to detect adulteration of EVOO with olive pomace [30].

After studying the characteristic bands of the scanned spectra, a PCA analysis was carried out to establish differences and similarities between adulterated and not adulterated oils when using each instrument (NIRscan nano or MicroNIR), different sample cups and scanning mode (transmittance or reflectance). After applying different math pretreatments to highlight differences between those adulterated oils and pure olive oils the PCA analysis was carried out. As can be seen in Fig. 7, after applying the following math pretreatment, 2<sup>nd</sup> SG derivative plus SNV, to spectra collected with both instruments, PCA results showed that: i) pure olive oils are clearly separated from adulterated samples, but ii) when using MicroNIR this statement cannot be established, because EVOO is the only oil that can be distinguished from adulterated samples.

After confirming differences between spectra data collected with both instruments. The next step was to evaluate the possibility of developing a quantitative procedure to establish the percentage of adulteration using both NIR instruments. As detailed in the Material and Methods section a PLS model was developed using spectra and reference data (percentage and adulteration type). Math pre-treatments were evaluated to determine the most accurate statistics for each model. These pre-treatments were based on scatter correction (SNV), and first or second SG derivatives. Table II summarizes the best model obtained for each instrument and oil used to adulterate olive oils. This table shows also the most relevant statistics when developing calibration models, the coefficient of determination of calibration, cross-validation, and standard error of calibration (*SE*).

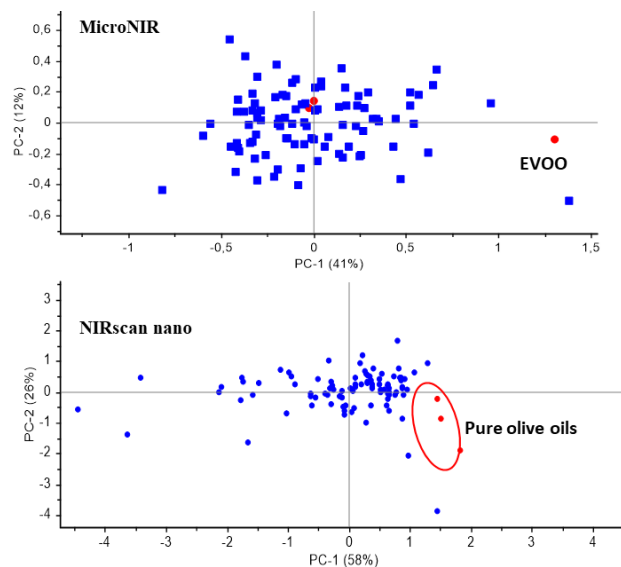


Fig. 7. Principal Component Analysis of spectra dated collected with both instruments. (EVOO: Extra Virgin Olive Oil).

TABLE II. CALIBRATION STATISTICS TO DETERMINE THE LEVEL OF ADULTERATION OF OLIVE OIL SAMPLES.

Adulteration oil	Math Pretreatment		Instrument	$R^2$	SE
Flax	1 4 4 SG SNV	CAL	NIRscan nano	0.778	2.9
		CV		0.747	3.1
	2 4 4 SG SNV	CAL	MicroNIR	0.9699	1.0
		CV		0.9001	2.1
Sesame	1 4 4 SG SNV	CAL	NIRscan nano	0.839	2.8
		CV		0.681	4.0
	2 4 4 SG SNV	CAL	MicroNIR	0.9699	1.0
		CV		0.9001	2.1
Sunflower	1 4 4 SG SNV	CAL	NIRscan nano	0.921	2.3
		CV		0.681	4.8
	2 4 4 SG SNV	CAL	MicroNIR	0.8437	2.8
		CV		0.5983	4.4

Results showed that it is not possible to establish a criterion to select one or another instrument. NIRscan Nano achieved better results than MicroNIR when calibration  $R^2$  values are considered. However, SE is lower when working in reflectance mode with the MicroNIR instrument. Although these are preliminary results, and more samples and increasing variability in the database should be included, both instruments can offer a non-invasive, and real-time analysis alternative to detect fraud in olive oils. From the point of view of cost NIRscan Nano has lower cost than MicroNIR.

## V. CONCLUSION

After testing two different NIR portable instruments with transmittance and reflectance scanning modes, MicroNIR and NIRscan Nano, it was possible to establish that, clear differences were observed, between spectra collected in Transmittance and Reflectance. Moreover, a qualitative assay (PCA) to distinguish adulterated or pure olive oils, was only possible when scanning samples with NIRscan nano in transmittance mode. However, focusing on quantitative calibration models, no significant differences were found when using one or the other instrument. Increasing the variability of the samples, including type and adulteration percentage would be an alternative to improve and validate these results. From the point of view of cost, NIRscan Nano EVM provides high performance and lower cost using a larger single-point 1-mm detector than a linear array with very small pixels.

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