Non-destructive analysis of intact olive fruits by near infrared spectroscopy: prediction of fat yield and acidity

Juan M. González, Mª Dolores Pérez-Marín, Ana Garrido-Varo and José E. Guerrero

Faculty of Agriculture and Forestry Engineering, ETSIAM, University of Córdoba, Apdo 3048, 14080 Córdoba, Spain. pa2pemad@uco.es

Introduction

Nowadays, in the olive oil sector, there is an increasing interest by all the aspects regarding the quality and organoleptic characteristics of the final product obtained. This fact is closely related with the characteristics and quality of the incoming raw material. Therefore, olive oil processing plants are increasing the quality control of the olive fruits used as raw material to produce virgin olive oil.

Traditionally, olive oil processing plant paid the olives to the farmer by weight of product, regardless of their quality. Nevertheless, some times ago the olive payment became to carry out according to the fat yield of the product and more recently according to the acidity content too. Free acidity and fat yield are the most relevant chemical parameters for a quick and reliable quality control and payment of olive fruits at the entry of the olive oil mill plant.

To give an answer to the olive oil sector demand, it's necessary to have analytical methods that be fast, versatile, economic and accurate. Near infrared (NIR) spectroscopy could be an alternative to the traditional physicochemical methods used for olive payment.

A previous work, presented at the 10th International NIR Conference held in Korea,[1] demonstrated the possibilities of near infrared spectroscopy for a non-destructive screening of olive fruits in the framework of breeding programmes. The present work tries to demonstrate the feasibility of NIRS to predict fat yield and acidity on intact olive fruits samples.

Material and methods

Sample set

A total of 76 olive samples of different varieties (*Picual, Picudo and Hojiblanca)*, from the 1999-2000 harvest, were taken to an olive oil processing plant.

Collecting spectra

The samples were scanned in a NIR diode-array spectrophotometer (Perten DA-7000 Flexi-Mode), working in reflectance mode in the spectral range between 400 to 1700 nm (at 5 nm intervals). Intact olive samples were analysed in the "Down-View-Rotating" mode, using the 12.7cm diameter open cup. Spectra were recorded with the Simplicity software.

Reference data

The reference data for fat yield and acidity were obtained by nuclear magnetic resonance (NMR) and a titration method, respectively. Values were expressed in an "as is" basis.

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Calibration development

Spectroscopic and chemical data were subjected to chemometric treatment using the software WinISI ver. 1.04 (Infrasoft International, Port Matilda, PA, USA). A modified partial least squares method was used to obtain regression equations for the two analytical parameters. All equations were obtained using the spectral region between 450 to 1650 nm, the standard normal variate and detrending methods for scatter correction and different derivative mathematical treatments. The statistics used to select the best equations were the coefficient of determination (r^2), the standard error of cross validation (*SECV*) and the ratios *RPD* (*DT*/*SECV*) and *RER* (Range/*SECV*).[2,3]

Results and discussion

Table 1 show the characteristics of the calibration set, displaying the values of the mean, range, standard deviation and coefficient of variation for fat yield and acidity. These statistics show that the calibration samples exhibit a high variability for the two chemical parameters studied.

Table1. Calibration set characteristics.

Parámetro	*N*	Range	Mean	*SD*	*CV*
Fat yield (%)	76	21.78–41.95	29.92	5.05	16.91
Acidity (%)	76	0.33–5.06	1.43	1.20	83.91

Table2. Calibration set characteristics

Constituent	PLS	Mean	Range	*SD*	*SECV*	r^2	*RPD*	*RER*	*CV*
Fat yield %	10	29.59	22.69–41.01	4.66	**1.02**	**0.95**	4.57	17.96	3.45
Acidity (%)	3	1.13	0.33–3.72	0.89	**0.27**	**0.91**	3.70	12.56	23.89

The NIR calibrations obtained afforded a high degree of precision in predicting the fat yield and the acidity content in intact olives (Table 2). The coefficients of determination for cross-validation (r^2) were higher than 0.9 for these two parameters. Similarly, standard errors of cross-validation (*SECV*) showed low values. The low *SECV* values together with the high *SD*s and the broad range for the two chemical parameters studied are the main reasons to explain the high *RPD* and *RER* values obtained for both calibration equations.

For fat yield, the errors obtained were lower than those reported by Leon et al. (2.20%),[1] although in this case the values were expressed in a dry matter basis. Jiménez *et al*.[4] obtained equations to predict fat yield with a r^2 of 0.98and a SEP value slightly lower (0.8%) than the *SECV* value reported in Table 2. However, these results were obtained on olive cake, while in the present work the samples were analysed ungrounded or intact.

The analysis of the acidity by NIRS on olive fruits has not been previously reported. The wavelength range scanned by the PERTEN DA-700 (400 nm–1700 nm) it does not include the traditional double spike (1725, 1760 nm) characteristic of fats and oils spectra. Despite of that, accurate equations for the prediction of acidity, avoiding the extraction of the fat from the fruit, has been obtained on the present work. These results confirm, that other spectral bands, as the second

overtone near 1200 nm, are also of great value for the prediction of fats characteristics, as the acidity.

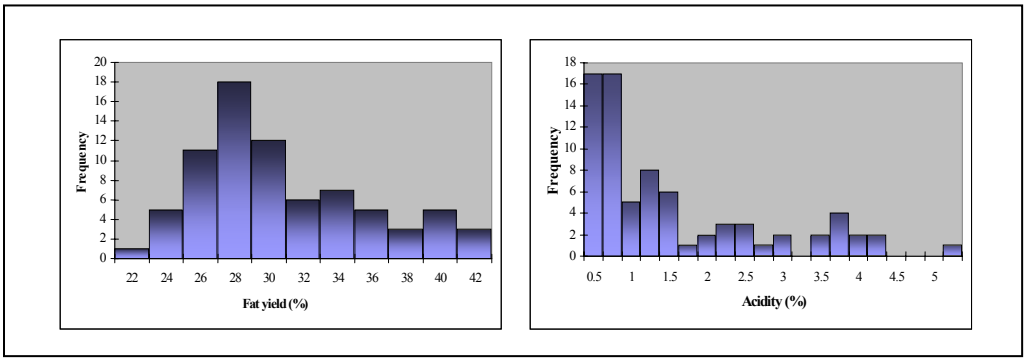

Figure1. Distribution of calibration samples for fat yield and acidity content.

Figure 1 illustrates sample distribution on the basis of the fat yield and acidity of the calibration set. The calibration statistics reported in Table 2 may be further improved by assembling a calibration set having more samples over and above the mean values for the two chemical constituents.

Conclusion

The results confirm the ability of NIR for the evaluation and classification of intact olive fruits according to their quality and for payment to olive oil producers.

References

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