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Analytical Methods

Near infrared spectroscopy (NIRS) for on-line determination of quality parameters in intact olives

Lourdes Salguero-Chaparro [a,*], Vincent Baeten [b], Juan A. Fernández-Pierna [b], Francisco Peña-Rodríguez [a]

[a] IFAPA "Alameda del Obispo", Avd. Menéndez Pidal s/n, 14004 Córdoba, Spain
[b] Walloon Agricultural Research Centre (CRA-W), Valorisation of Agricultural Products Department, Food and Feed Quality Unit (U15), 'Henseval Building', Chaussée de Namur 24, 5030 Gembloux, Belgium

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ABSTRACT

The acidity, moisture and fat content in intact olive fruits were determined on-line using a NIR diode array instrument, operating on a conveyor belt. Four sets of calibrations models were obtained by means of different combinations from samples collected during 2009–2010 and 2010–2011, using full-cross and external validation. Several preprocessing treatments such as derivatives and scatter correction were investigated by using the root mean square error of cross-validation (RMSECV) and prediction (RMSEP), as control parameters. The results obtained showed RMSECV values of 2.54–3.26 for moisture, 2.35–2.71 for fat content and 2.50–3.26 for acidity parameters, depending on the calibration model developed. Calibrations for moisture, fat content and acidity gave residual predictive deviation (RPD) values of 2.76, 2.37 and 1.60, respectively. Although, it is concluded that the on-line NIRS prediction results were acceptable for the three parameters measured in intact olive samples in movement, the models developed must be improved in order to increase their accuracy before final NIRS implementation at mills.

1. Introduction

The olive tree (*Olea europaea* L.) is one of the most ancient domestic fruit trees cultivated in the Mediterranean regions and has an important economic impact mainly due to the production of olive oil. Virgin olive oil is obtained from olive fruits using only mechanical means and without the application of any chemical treatment. The quality of a virgin olive oil can be affected by several factors, such as the agronomic techniques used, the seasonal conditions and the ripening stage among others. In order to assure the correct quality of this oil, characterisation and quality control of the raw materials is required at the entrance of the olive processing plants (mills) as well as before starting olive oil extraction process, because the quality of the olives can be changed during the storage.

From the mill industrial point of view, the three main quality parameters are the oil content and moisture of the fruit and free acidity of olive oil. The knowledge of these parameters allows to determine the payment to the growers and the final destination of the olives (acceptability for milling or rejection and storage monitoring). Traditionally, these parameters have been estimated by analytical methods in chemical laboratories, such as Soxhlet and Foss-let (Núñez et al., 2005) for the determination of the oil content, as the gravimetric method for the determination of moisture and as the acid–base titrations for the determination of free acidity. In most of cases, these methods are tedious, destructive, time consuming, require considerable sample manipulation, especially for large sample sets and also organic solvent consuming (Muik, Bernhard, Molina-Diaz, & Ayora-Cañada, 2003). Moreover they do not allow obtaining a quick response in oil content and moisture determinations, since require a prior milling of the olives and a subsequent drying of the paste obtained. Therefore, in order to carry out an effective quality control and characterisation of the receiving olives, fast, versatile and non destructive methods are necessary for the instant measurement of all these parameters.

In this context, near infrared spectroscopy (NIRS) has been one of the most important techniques to be used in the food and feed laboratories in the last years. NIRS already plays an important role due to its ability for rapid and non-destructive quantitative and qualitative analysis of a high variety of products and constituents in food/feed industry (Fernández Pierna, Baeten, & Dardenne, 2006; Murray, Aucott, & Pike, 2001; Osborne, Fearn, & Hindle, 1993). As far as olives are concerned, near infrared applications regarding olive pomaces, pastes and oils composition have been reported many times and a survey of these studies has been given (Armenta, Moros, Garrigues, & De La Guardia, 2010). However, to our knowledge, few papers in the literature have been dealing with the use of NIR spectroscopy in intact olive fruits.

* Corresponding author.
E-mail address: salg_lou@hotmail.com (L. Salguero-Chaparro).

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The first applications in intact olives were developed with a Perten diode array reflectance spectrometer. In this way, León, Rallo, and Garrido (2003) and León, Garrido-Varo, and Downey (2004) carried out the analysis of intact olive samples in order to determine the oil content and moisture of olives as well as fatty acids' composition of olive oil by using NIRS. Evaluation of the use of this technology for the determination of acidity and oil content in intact olive fruits was also carried out by González, Pérez-Marín, Garrido-Varo, and Guerrero (2004). Similar studies were done by Morales-Sillero et al. (2011) that confirmed that NIRS can be used as a useful technique to assess the properties of intact olives. For that, spectral data were recorded using a Foss-NIRSystems 6500 SY-II monochromator instrument. Recent advances in NIRS technology have allowed the development of field portable instruments, that enable collect spectral information directly on the fruit on the tree (Cayuela, Garcia, & Caliani, 2009), allowing a more rapid and efficient analysis (Cayuela & Pérez-Camino, 2010; Gracia & León, 2011).

However, until now, few papers dealing with on-line process control using NIRS in the olive mill industry have been published. One major reason is probably the lack of suitable sensing devices, which are required to be at the same time robust and non-destructive (Hildrum, Nilson, Westad, & Wahlgren, 2004). One of the first preliminary studies of the viability of NIRS on-line methods was performed by Hermoso, Uceda, García-Ortiz, Jiménez, and Beltrán (1999). In this paper, the technique was used for the measurement of the oil content and moisture in olive pomace from a two-year campaign. The NIRS equipment (MM55E Infrared Engineering, Maldon, Essex, UK) was installed at the decanter olive pomace passage. In another study, Jiménez-Márquez, Molina-Díaz, and Pascual-Reguera (2005) applied near infrared transmittance spectroscopy to on-line control quality and characterisation of virgin olive oils. PLS models were developed for acidity value, bitter taste and fatty acid's composition. The use of NIRS was also investigated by Gallardo-González, Osorio-Bueno, and Sánchez-Casas (2005) for determining in real time the moisture and oil contents of olive pastes and the resulting olive wastes generated in the 2-phase oil extraction process. All the studies mentioned here indicated that NIRS gives favourable performance parameters compared with the analytical techniques for on-line analysis in olive pomace, olive pastes and olive oils. However, no scientific information has hitherto been available regarding the viability of NIR spectrometers for on-line quality control in intact olives.

In the present study, a reflectance instrument for NIRS measurements (CORONA 45 Vis/NIR, Carl Zeiss Jena GMBh, Jena, Germany) with a diode array detector was used on a conveyor belt set for the on-line determination of the oil content, moisture and free acidity parameters in intact olive fruits.

2. Materials and methods

2.1. Olive samples

Olives (*O. europaea* L.) from more than 50 different varieties have been used in this study. 38% of the olives were from the 'Picual' cultivar, which is the variety most extended in Spain, and especially in Andalusia. The olives were gathered during the harvesting time (October–March) of two olive crop seasons (2009–2010 and 2010–2011), mainly from the region of Córdoba and Jaén, but also from other regions of Andalusia.

The olives were harvested manually, and leaves and damaged olives were removed. After, the olives were stored in a refrigeration chamber at 5 °C and 90% relative humidity until their chemical and spectroscopic analyses.

2.2. On-line NIR analysis

This work was performed at the laboratory of the Food and Postharvest Technology Area at the IFAPA "Alameda del Obispo" (Cordoba, Spain), where a process line for the project was built, as shown in Fig. 1. A set of four conveyor belts were aligned and disposed each one with respect to the previous one for guarantee receiving olives in a continuous way. On a base placed next to the largest and flat conveyor belt, the computer and NIRS equipment were installed.

After a sample stabilisation for 24 h at laboratory temperature (24 °C) using an air conditioner equipment, since this factor can strongly affect background levels during spectrum acquisition (Williams, 2001), the olives were transferred to a conveyor belt of 2.8×2.10 m, over which the NIRS instrument was mounted on the base to get on-line measurements.

Spectral Vis/NIR measurements were recorded using a Zeiss Corona 45 Vis/NIR diode-array spectrophotometer (Carl Zeiss, Jena, Germany). This instrument is fast and robust, without moving parts and provides spectral information in the wavelength range between 380 and 1690 nm, at a resolution of 2 nm. It is equipped with a silicon diode array (Hamamatsu S 3904) for the wavelength region 380–950 nm and an InGaAs array for the range 950–1690 nm. Instrument references were achieved with external black and white standards.

After stabilising the instrument during 1 h, the measurements in the olive samples (of approximately 10–20 kg) were performed. Previous to the measuring of the samples, a complete optimisation of the system (not shown here) has been performed (Salguero-Chaparro, Baeten, Abbas, & Peña-Rodríguez, 2012). This system optimisation allowed defining some important parameters as the distance from sensor to surface of the sample on the conveyor belt, which was fixed to 13 mm, the speed of conveyor belt (0.1 ms^{-1}) and the thickness of layer, which was kept constant.

Spectra were acquired with an integration time of 5 s and the final spectra corresponded to the average of 10 scans for each sample. A total of thirty spectra were acquired per olive fruit sample and averaged.

2.3. Software

Instrument control and initial spectral manipulation were carried out using CORA software v.3.2.2. (Carl Zeiss, Inc., Thornwood, NY, USA) and WinISI III software v.1.50 (Infrasoft International, Port Matilda, PA, USA).

Spectral data were processed using the Matlab software package version 7.0 (The Mathworks, Inc., Natick, MA, USA). The PLS

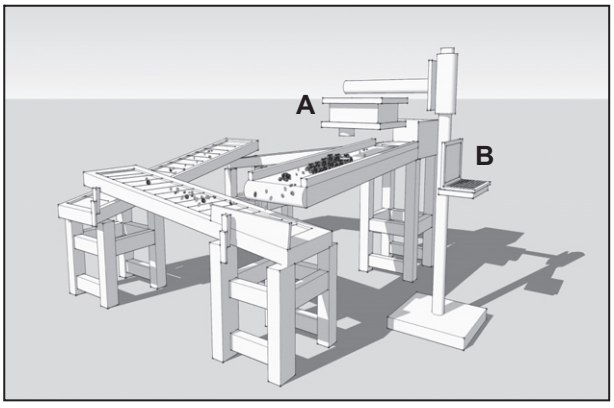

Fig. 1. Scheme of CORONA 45 Vis/NIR instrument on the process line. (A): NIR equipment and (B): computer.

Toolbox (version 4.11, EigenvectorResearch, Inc., Manson, WA, USA) was used for principal component analysis (PCA) and PLS regression.

2.4. Reference analysis

After spectral acquisition, the olive oils were extracted using an Abencor laboratory mill (MC2 Ingeniería y Sistemas, S.L. Seville, Spain), simulating commercial oil-extraction systems. The process consisted of the following steps: 1000 g of olives were ground in a hammer mill. Then, the crushed sample was divided into two parts. One was used for the determination of the moisture and the oil content and the other part, approximately 700 g, was beaten in a thermo beater with talc and then centrifuged (two rounds of 60 s each at 3000 rpm) to obtain the oil for free acidity determination (Frías et al., 1991).

Around 25 g of the olive paste obtained was placed in capsules to determine moisture and dried for 18 h in an oven at 105 °C. Olive moisture was gravimetrically determined and expressed as percentage of fresh weight (AENOR, 1973). Determination of free acidity value was carried out according to the analytical method described in Regulation EEC/2568/91 of the European Union Commission (EC, 2008) and expressed as oleic acid percentage (%w/w). Finally, Soxhlet extraction was carried out to determine the oil content from the dried material of each sample using 180 ml of *n*-hexane as solvent (UNE 55030) (AENOR, 1961).

Fig. 2 shows the distribution of the obtained values for each parameter determined by reference analysis. Table 1 shows the mean, standard deviation (SD) and range of the figures obtained by reference values for the samples used in this study for the three constituents analysed (free acidity, moisture and oil content).

3. Results and discussion

Water and oil are the major components of the pulp and the skin in the olive fruit. At the usual harvest time for oil production, the pulp (which accounts for approximately 75% of the total weight of the fruit) has about 30–60% water and 20–35% oil. In the skin water represents 30% and oil 27%. Sugars (3%) and polysaccharides (mainly cellulose, hemicellulose and lignin) (4%) are also present in the pulp (Beltrán, Uceda, Hermoso, & Frías, 2004). Spectra for samples of intact olives acquired with the CORONA 45 Vis/NIR instrument presented in the visible region two particularly noticeable peaks around 670 and 446 nm due to the chlorophyll and carotenoids pigments present in the fruits, respectively (Chaoyang, Zheng, Quan, & Wenjiang, 2008). Furthermore, peaks located at 970 nm and around 1200 nm supplies information related to the second overtone of the O–H stretching vibration of water and to the C–H second overtone of fat, respectively (Büning-Pfaue, 2003; Williams & Norris, 1987). Finally, absorption located around the 1440 nm has been linked in olive products to the O–H stretch first overtone of water (Jiménez, Izquierdo, Rodríguez, Dueñas, & Tortosa, 2000; Kavdir, Buyukcan, Lu, Kocabiyik, & Seker, 2009).

After the visual observation of the characteristic peaks in the spectra, a principal component analysis (PCA) was done. This algorithm is an explorative data technique that can be used to determine natural clusters and groups with similar characteristics and to give an insight of possible outliers (Massart, Vandeginstem, Deming, Michotte, & Kaufman, 1988). Here, PCA has been applied in order to check the distribution of the samples in the data set conformed by the two years (2009–2010 and 2010–2011). Previously to the PCA, a multiplicative scatter correction (MSC) treatment was applied. MSC is designed to remove from reflectance spectra at least some of the large amount of variability that may be caused by scattering effects (Fearn, Riccioli, Garrido-Varo, & Guerrero-Ginel, 2009).

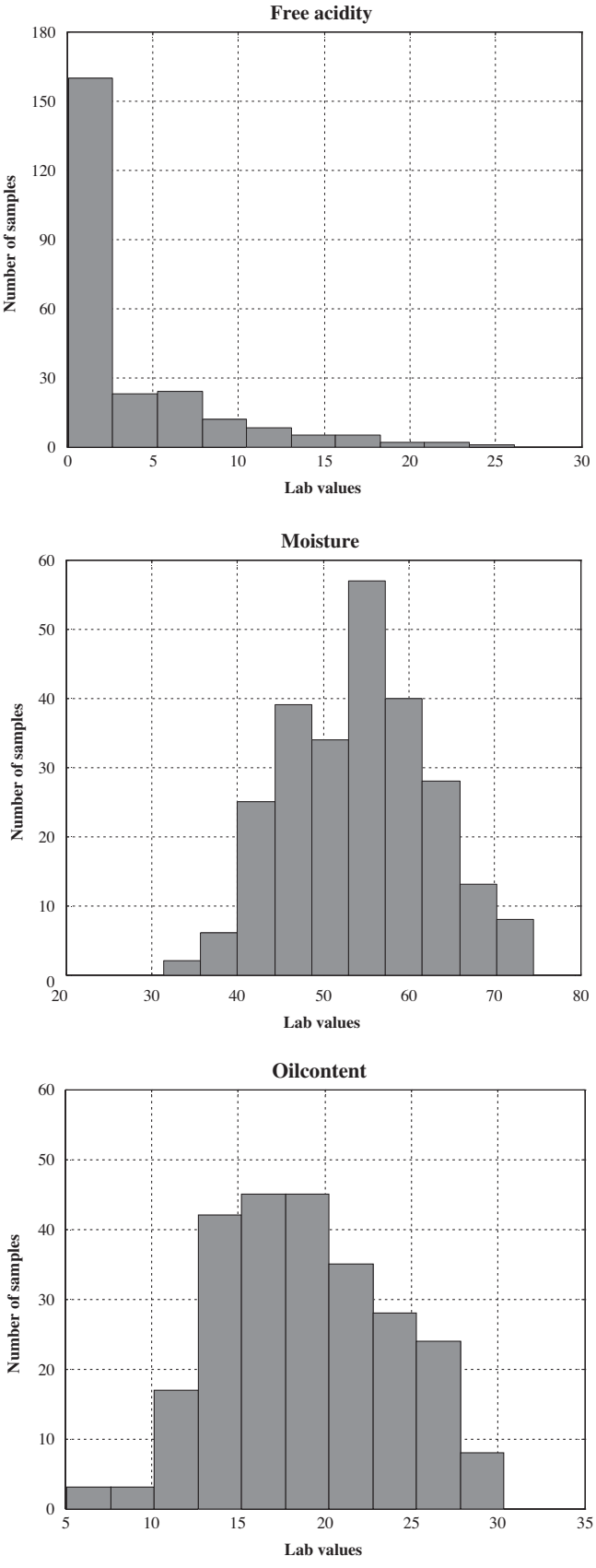

Fig. 2. Frequency histograms of reference lab values for free acidity, fat content and moisture.

Table 1
Chemical characterisation of the olive samples for building PLS models.

	Number of samples	Mean	SD	Range
Free acidity (% oleic acid)	242	3.31	4.85	0.09–26.06
Moisture (%)	252	54.25	8.29	31.42–74.55
Oil content (%)	250	18.85	4.92	5.11–30.34

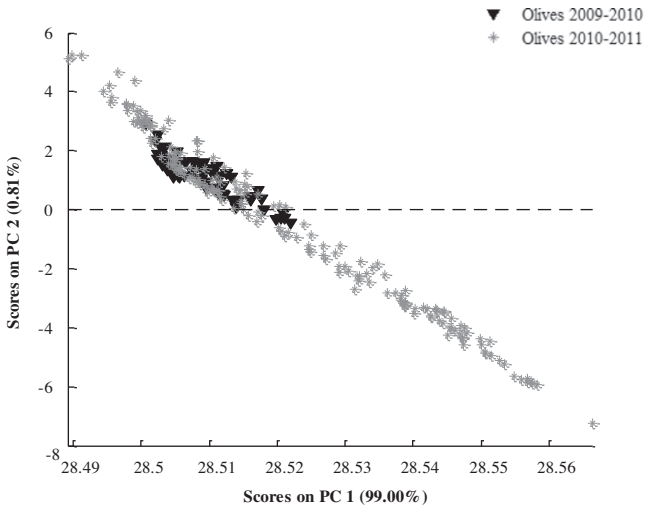

Fig. 3. Principal Components Analysis (PC1 and PC2), after a multiplicative scatter correction for samples from 2009 and 2010 years.

The PCA plot of the samples is shown in Fig. 3, where the first two PCs explained 99.81% of the total variation in the spectral matrix. As can be observed, although a clear level of clustering is observed among samples of the first campaign (2009–2010), olives harvested during the first year are included in the space comprised by the samples of the second campaign (2010–2011), showing no separation between both years.

Once the PCA was applied (no outliers were detected), quantitative calibrations were developed for predicting oil content, moisture and free acidity in intact olives, using Partial Least Squares (PLS) as regression method. The main idea of PLS regression is to get as much concentration information as possible into the first few loading vectors using both reference data (chemical, physical, etc.) and spectral information. In this study, leave-one-out cross-validation (LOOCV) and external validation were used for establishing and validating calibration models. For each iteration of LOOCV, one sample was left out and the multivariate model was constructed by the rest of the samples. The process was repeated until all the samples were used once in the validation set.

Saeys, Darius, and Ramon (2004) established several levels of prediction accuracy for a heterogeneous material based on the RPD values. In this way, an RPD value between 1.5 and 2.0 indicates a possibility to distinguish between high and low values, while a value between 2.0 and 2.5 makes approximate quantitative predictions possible. Finally, an RPD value between 2.5 and 3.0 reveals a good prediction of this calibration.

For the determination of these quality parameters (oil content, moisture and free acidity), four different strategies were developed using different calibration and external validation sets:

- *Strategy 1:* Calibration model with samples collected in 2009 and validation with samples collected in 2010.
- *Strategy 2:* Calibration model with samples collected in 2010 and validation with samples collected in 2009.
- *Strategy 3:* Calibration model with all samples collected in 2009 and 70 samples collected in 2010 and validation with the rest of samples collected in 2010.
- *Strategy 4:* Combination of the 2009 and 2010 data sets (60% samples for calibration model and 40% for validation model, selected by Duplex algorithm).

For each strategy studied, the results of the PLS models for free acidity, moisture and oil content are summarised in Table 2.

As it can be observed in Table 2, prediction models for all parameters provided better results in the strategy 2 than in strategy 1, where R^2 showed values higher than 0.70 in all cases and

Table 2
Results of the PLS models for free acidity, moisture and fat content determination in intact olives.

	Strategy 1	Strategy 2	Strategy 3	Strategy 4
Free acidity				
Pretreatment	SNVDT (15,2,2)	MSC (9,2,1)	SNVDT (None)	MSC (None)
No. latent variables	6	7	8	8
R^2	0.41	0.72	0.74	0.72
RMSEC (%)	2.39	2.16	2.53	2.44
RMSECV (%)	3.41	2.50	2.94	2.79
RMSEP (%)	4.67	3.12	2.53	2.70
RPD	1.02	1.63	1.60	1.81
Moisture				
Pretreatment	SNVDT (15,2,2)	SNVDT (15,2,2)	SNVDT (None)	SNVDT (15,2,1)
No. latent variables	6	6	7	6
R^2	0.76	0.87	0.87	0.88
RMSEC (%)	2.02	2.29	2.95	2.57
RMSECV (%)	2.65	2.54	3.26	3.19
RMSEP (%)	5.48	4.89	2.98	3.30
RPD	1.56	0.98	2.76	2.56
Oil content				
Pretreatment	MSC (15,2,2)	SNVDT (None)	SNVDT (None)	SNVDT (None)
No. latent variables	5	7	8	8
R^2	0.52	0.75	0.79	0.79
RMSEC (%)	2.05	2.18	2.34	2.22
RMSECV (%)	2.71	2.35	2.66	2.53
RMSEP (%)	4.09	3.05	2.15	2.36
RPD	1.27	1.08	2.37	2.05

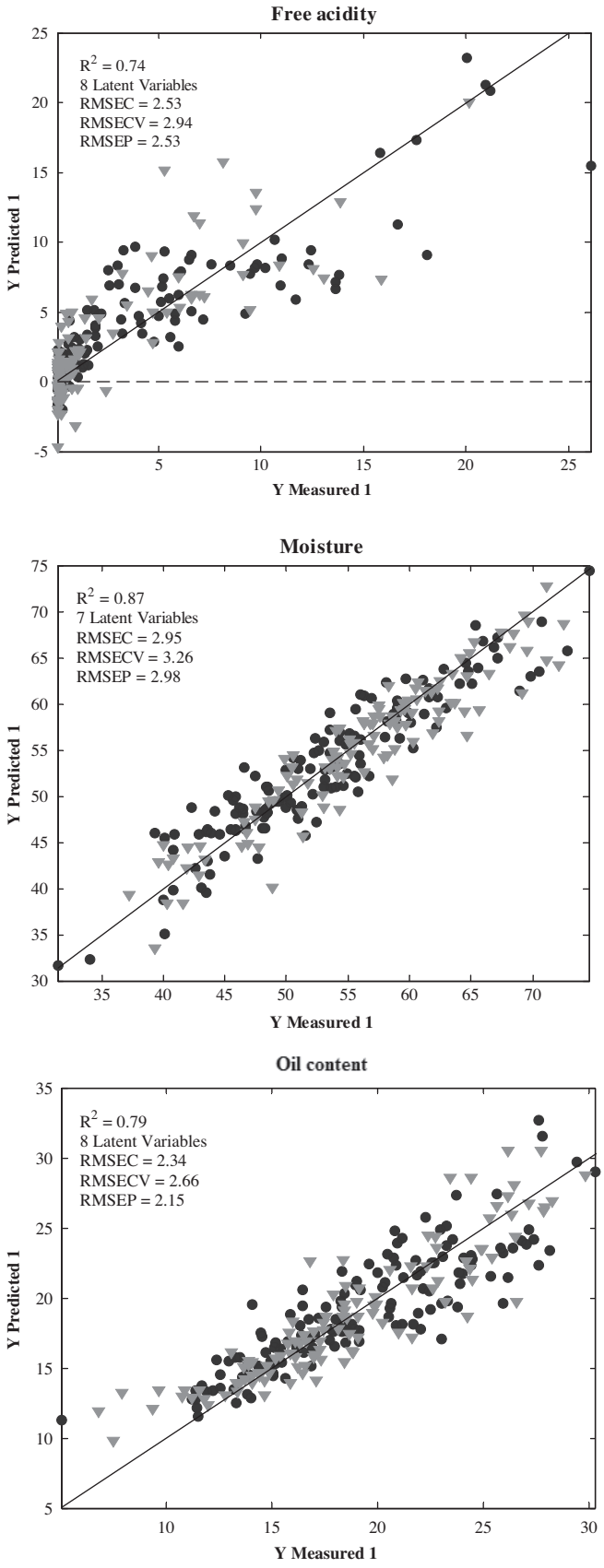

Fig. 4. NIR-predicted data versus reference data for the external validation sets for acidity, moisture and fat content in the strategy 3.

RMSEP values of 3.12%, 4.89% and 3.05% for free acidity, moisture and oil content, respectively. However, these results were expected due to the fact that the variability included in the first harvest period was much lower than the variability in the second campaign. During the first harvest period, bad weather conditions (constant rains) avoided an adequate and diverse collection of olives, which could be representative of the whole variability of a normal campaign, in terms of geographic origin of the samples, composition, agroclimatic conditions (rain, temperature, hours of sun, etc.) and other factors such as the variety.

Although it is not possible to establish a clear advantage from using strategy 3 or strategy 4, because of the results presented similar statistics values for the three olive quality parameters, values obtained in strategy 3 were slightly better than those obtained in strategy 4 in terms of R^2 and RMSEP. R^2 values of 0.74, 0.87 and 0.79 were obtained for free acidity, moisture and oil content, respectively and a RMSEP value lower than 3% for all cases in strategy 3. In this case, the external validations showed good predictions for moisture, with an RPD value of 2.76 and for oil content (RPD = 2.37) and also acceptable results for free acidity parameter (RPD = 1.60). NIR-predicted data versus reference data for the external validation sets for each parameter of the strategy 3 is shown in Fig. 4.

As indicated earlier, the literature provides no data regarding the prediction of oil content, moisture and free acidity of intact olive fruits using a diode array instrument in dynamic conditions, so the results obtained may only be compared with those reported by other authors using at/off-line NIR instruments. Nevertheless, direct comparisons are not relevant here since the sampling errors are not the same in both types of analysis. This can be explained by the fact that larger batches are analysed during the acquisition of the spectrum in movement (on-line) compared to the relatively small sample used for the chemical analysis of reference. These samples taken for reference analysis cannot represent the true average of the whole batch and therefore, the sampling error will be probably much higher in these on-line measurements (Isaksson, Nilsen, Tøgersen, Hammond, & Hildrum, 1996).

Results for oil content obtained in this study compare quite well with the accuracy of equations reported by Cayuela and Pérez-Camino (2010) (R^2 = 0.78; RPD = 2.77). However, for oil content Gracia and León (2011) obtained a higher R^2 value (0.89) and lower RMSECV value (1.99%). On-line equations for moisture developed with the diode array device showed R^2 and RPD values slightly higher than those observed in Morales-Sillero et al. (2011) (R^2 = 0.86; RPD = 2.70). Nevertheless, Cayuela et al. (2009) recorded a quite low RMSEP value (1.52%) for moisture content. Finally, the results obtained for free acidity showed a much lower RPD and R^2 values than those obtained by Cayuela and Pérez-Camino (2010) (2.67 and 0.80, respectively).

4. Conclusions

The study reports here an on-line application of NIRS technology as a method to monitor and control the olive quality parameters (oil content, moisture and free acidity), without the need for milling the fruits, allowing in this way, provide real time information at the reception in the mill, about the quality and composition of the olives that will be used in the olive oil extraction process.

The results obtained may suggest the viability of diode array instrument for use in real on-line industrial environments. Nevertheless, further studies with larger data sets are necessary before the definitive implementation of these calibrations at mill industry level in order to improve the robustness of calibration models to become more stable for on-line monitoring of intact olives.

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References

AENOR, Asociación Española de Normalización y Certificación. (1961). *Cuerpos Grasos. Determinación del contenido en materia grasa total de la aceituna*. Norma UNE 55030, Madrid, España.

AENOR, Asociación Española de Normalización y Certificación. (1973). *Materias Grasas. Humedad y materias volátiles (Método de la estufa de aire)*. Norma UNE 55-020-73, Madrid, España.

Armenta, S., Moros, J., Garrigues, S., & De La Guardia, M. (2010). The use of near-infrared spectrometry in the olive oil industry. *Critical Reviews in Food Science and Nutrition, 50*, 567–582.

Beltrán, G., Uceda, M., Hermoso, M., & Frias, L. (2004). Maduracion. In D. Barranco, R. Fernández-Escobar, & L. Rallo (Eds.), *El cultivo del olivo* (5th ed.. Madrid, Spain: Junta de Andalucia, Grupo Mundi-Prensa.

Büning-Pfaue, H. (2003). Analysis of water in food by near infrared spectroscopy. *Food Chemistry, 82*, 107–115.

Cayuela, J. A., Garcia, J. M., & Caliani, N. (2009). NIR prediction of fruit moisture, free acidity and oil content in intact olives. *Grasas y Aceites, 60*, 194–202.

Cayuela, J. A., & Pérez-Camino, M. C. (2010). Prediction of quality of intact olives by near infrared spectroscopy. *European Journal of Lipid Science and Technology, 112*, 1209–1217.

Chaoyang, W., Zheng, N., Quan, T., & Wenjiang, H. (2008). Estimating chlorophyll content from hyperspectral vegetation indices: Modeling and validation. *Agricultural and Forest Meteorology, 148*, 1230–1241.

European Commission (EC) regulation 1991R2008 amending Regulation (ECC) No 2568/91 on the characteristics of olive oil and olive-pomace oil and on the relevant methods of analysis. (2008). Office for Official Publications of the European Communities, Luxembourg.

Fearn, T., Riccioli, C., Garrido-Varo, A., & Guerrero-Ginel, J. E. (2009). On the geometry of SNV and MSC. *Chemometrics and Intelligent Laboratory Systems, 96*, 22–26.

Fernández-Pierna, J. A., Baeten, V., & Dardenne, P. (2006). Screening of compound feeds using NIR hyperspectral data. *Chemometrics and Intelligent Laboratory Systems, 84*, 114–118.

Frías, L., García-Ortiz, A., Hermoso, M., Jiménez, A., Llavero Del Pozo, M.P., Morales, J., Ruano, T., & Uceda, M. (1991). *Analistas de laboratorio de almazara. Informaciones Técnicas*, 6/91. Junta de Andalucía, Sevilla, Spain.

Gallardo-González, L., Osorio-Bueno, E., & Sánchez-Casas, J. (2005). Application of near infrared spectroscopy (NIRS) for the real-time determination of moisture and fat contents in olive pastes and wastes of oil extraction. *Alimentación Equipos y Tecnología, 24*, 85–89.

González, J.M., Pérez-Marín, M.D., Garrido-Varo, A., & Guerrero, J.E. (2004). Non-destructive analysis of intact olive fruits by near infrared spectroscopy: Prediction of fat yield and acidity. In *Near Infrared Spectroscopy: Proceedings of the 11th International Conference* (pp. 373–375). NIR Publications.

Gracia, A., & León, L. (2011). Non-destructive assessment of olive fruit ripening by portable near infrared spectroscopy. *Grasas y Aceites, 62*, 268–274.

Hermoso, M., Uceda, M., García-Ortiz, A., Jiménez, A., & Beltrán, G. (1999). Preliminary results of NIR "on-line" measure of oil content and humidity in olive cakes from the two phases decanter. *Acta Horticulturae, 474*, 717–719.

Hildrum, K. I., Nilson, B. N., Westad, F., & Wahlgren, N. M. (2004). In-line analysis of ground beef using a diode array near infrared instrument on a conveyor belt. *Journal of Near Infrared Spectroscopy, 12*, 367–376.

Isaksson, T., Nilsen, B. N., Tøgersen, G., Hammond, R. P., & Hildrum, K. I. (1996). On-line, proximate analysis of ground beef directly at a meat grinder outlet. *Meat Science, 43*, 245–253.

Jiménez, A., Izquierdo, E., Rodríguez, F., Dueñas, J. I., & Tortosa, C. (2000). Determination of fat and moisture in olives by near-infrared reflectance spectroscopy. *Grasas y Aceites, 51*, 311–315.

Jiménez-Márquez, A., Molina-Díaz, A., & Pascual-Reguera, M. I. (2005). Using optical NIR sensor for on-line virgin olive oils characterization. *Sensors and Actuators, B, 107*, 64–68.

Kavdir, I., Buyukcan, M. B., Lu, R. F., Kocabiyik, H., & Seker, M. (2009). Prediction of olive quality using FT-NIR spectroscopy in reflectance and transmittance modes. *Biosystems Engineering, 103*, 304–312.

León, L., Garrido-Varo, A., & Downey, G. (2004). Parent and harvest year effects on near-infrared reflectance spectroscopic analysis of olive (*Olea europaea* L.) fruit traits. *Journal of Agricultural and Food Chemistry, 52*, 4957–4962.

León, L., Rallo, L., & Garrido, A. (2003). Analysis of intact olive fruit by near-infrared spectroscopy (NIRS): An useful tool in olive breeding programs. *Grasas y Aceites, 54*, 41–47.

Massart, D. L., Vandeginstem, B. G. M., Deming, S. N., Michotte, Y., & Kaufman, L. . *Chemometrics: A textbook* (Vol. 23). Elsevier Science Publisher (pp. 395–397).

Morales-Sillero, A., Fernandez-Cabanas, V. M., Casanova, L., Jimenez, M. R., Suarez, M. P., & Rallo, P. (2011). Feasibility of NIR spectroscopy for non-destructive characterization of table olive traits. *Journal of Food Engineering, 107*, 99–106.

Muik, B., Bernhard, L., Molina-Diaz, A., & Ayora-Cañada, M. J. (2003). Fourier transform Raman spectrometry for the quantitative analysis of oil content and humidity in olives. *Applied Spectroscopy, 57*, 233–237.

Murray, I., Aucott, L. S., & Pike, I. H. (2001). Use of discriminant analysis on visible and near infrared reflectance spectra to detect adulteration of fishmeal with meat and bone meal. *Journal of Near Infrared Spectroscopy, 9*, 297–311.

Núñez, N., Pérez, D., Salas, J., Gómez, L., Soto, J., García, J., Fernández, V.M. & Del Moral, R. (2005). Determinación instantánea mediante NIRS de los parámetros de calidad de la aceituna: Acidez, rendimiento graso y humedad. Foro de la Tecnología Oleícola y de la Calidad. XII Simposium Científico-Técnico, Expoliva 2005.

Osborne, B. G., Fearn, T., & Hindle, P. H. (1993). Introduction. In B. G. Osborne, T. Fearn, & P. H. Hindle (Eds.), *Practical NIR spectroscopy with applications in food and beverage analysis* (pp. 1–12). London, UK: Longman Scientific and Technical.

Saeys, W., Darius, P., & Ramon, H. (2004). Potential for on site analysis of hog manure using a visual and near-infrared diode array reflectance spectrometer. *Journal of Near Infrared Spectroscopy, 12*, 299–309.

Salguero-Chaparro, L., Baeten, V., Abbas, O., & Peña-Rodríguez, F. (2012). On-line análisis of intact olive fruits by vis-NIR spectroscopy: Optimisation of the acquisition parameters. *Journal of Food Engineering, 112*, 152–157.

Williams, P. C. (2001). Sampling, sample preparation and sample selection. In D. A. Burns & E. W. Ciurczak (Eds.), *Handbook of near infrared analysis* (2nd ed., pp. 307–350). New York, USA: Marcel Dekker.

Williams, P. C., & Norris, K. (Eds.). (1987). *Near infrared technology in the agricultural and food industries*. St. Paul, Minnesota: American Association of Cereal Chemists.