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NIR Spectroscopy for the Online Monitoring of Water and Olive Oil Content in Pomace during the Extraction Process

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Abstract: The main challenge of this scientific work was the implementation on an industrial olive oil extraction plant of an NIR device for the multispectral analysis of pomace to predict the percentage of humidity and oil contained in it. Subsequent to the implementation of the NIR device on the oil extraction line on the solid's outlet from the decanter, NIRS interaction measurements in the 761–1081 nm region were used to probe the pomace. NIRS calibration models for the prediction of water and oil content in the pomace were obtained and successfully tested and validated. The correlations of calibration results for oil and water content were 0.700 and 0.829, while the correlations of validation were 0.773 and 0.676, respectively. Low values of root mean square error were found for both the prediction and validation set. The results highlight the good robustness of an NIR approach based on a PLS calibration model to monitor the industrial olive oil process. The results obtained are a first step toward the large-scale implementation of NIR devices for monitoring pomace in oil mills. The possibility of knowing the oil lost in the pomace, moment by moment, would open a new frontier towards system control and the sustainability of the olive oil extraction process.

Keywords: in-line NIR; industrial instrumentation; olive oil process; PLS regression model; water/oil content

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1. Introduction

Virgin and extra virgin olive oil are obtained directly from olives and solely by mechanical means [1]. The olive oil extraction process comprises several steps, including the cleaning of the olives (leaf removal and washing), preparation of the olive paste, conditioning of the olive paste, the centrifugal separation of solids and liquids, and finally the centrifugal separation of the liquids [2,3]. The process is implemented by a sequence of machines arranged in series or in parallel.

Plant extractability (E), defined as the percentage ratio between the oil extracted and the oil contained in the olives, is the parameter used to evaluate the extractive performance of the plant [4]. The extractability generally varies from 80 to 90% of the total oil content in olives [5–8]. Ten to twenty percent of the oil contained in olives is lost in the pomace; this occurs because the oil in the olive paste is only partially free to escape, and part of it remains in the unbroken cells, is trapped in the tissues of the cytoplasm, or is emulsified in the aqueous phase [9–11].

Many efforts have been made by research institutes, plant manufacturers, and plant managers to identify the best conditions to both increase the quality of the oil [12–16] and reduce oil losses in pomace. Considering the process phases described above, those that most influence the extractability of the plant are the preparation and conditioning of the olive paste and the solid–liquid separation carried out by the decanter. It has been demonstrated that using a destoning machine leads to a slight decrease in the extractability

of the oil [17–19]. It has also been demonstrated that the type of crusher, diameter of the holes in the grid, malaxation times, and temperatures can influence extractability [20]. In the end, the adjustment of the decanter can have a strong impact on the amount of oil extracted [4,5,21,22].

It has been demonstrated that when the process is strictly controlled in each phase and when the decanter is adjusted optimally, the extractability can even exceed 90%, peaking at 92.5%, as reported in [23].

To improve the olive oil process by reducing the oil losses in pomace, the addition of technological supports such as talc or enzymes can, respectively, promote the breakdown of oil-in-water emulsions [24–26] and the breakdown of cellular structures in pulp [27,28], with a consequent increase in oil extractability and decrease in pomace oil loss.

Recently, different innovative technologies based on the use of microwaves, ultrasound, and pulsed electric fields have been introduced in the extraction process, often increasing the extractability of the oil [29–31].

Knowing the percentage of residual oil in pomace is considered a crucial aspect for optimizing extraction plants, which is influenced by the choice of machines and their adjustment.

Currently, the official method to determine the amount of oil lost in pomace requires a time-consuming drying step, followed by solvent extraction [32]. This technique, although accurately returning the humidity and oil content percentage of the sample analyzed, takes more than one day. Considering the long time necessary to obtain the result, this type of analysis is used for machine optimization during the testing phase, at the beginning of each harvest season, or periodically during harvesting to evaluate plant performance.

However, to achieve real-time feedback and control, mainly of the decanter, but also of the entire extraction plant, continuous measurement of the water and oil content of the pomace discharged from the decanter is crucial. These measurements make it possible to evaluate whether the decanter or the plant are meeting optimal extraction conditions [33].

In recent years, it has been demonstrated that the use of near-infrared (NIR) spectrometry allows for the measurement of the oil and water content in olives and pomace with satisfactory results in a very short time. Benchtop NIR technology was used to predict the moisture content and oil content of pomace, and good predictive models were found, as reported in [6,34].

Using only benchtop NIR technology in [33], spectrophotometric models suitable for the rapid and accurate measurement of the oil and water content in olive pomace were developed. The accuracy of the models was 3.7% (±0.5) and 2.5% (±0.5) for oil content in pomace on a wet and dry basis, respectively.

A preliminary study [35] was carried out to verify the applicability of portable vis/NIR spectroscopy devices to predict the oil content in pomace during the milling process. Strong predictive models were developed and applied in an online system to monitor oil loss and perform a feed-forward control to improve the process efficiency.

The studies mentioned above have demonstrated the feasibility of using near-infrared spectrometry analysis by using a laboratory instrument or a portable instrument to predict water and oil content in pomace.

Currently, despite research and application efforts, NIR technology is used in mills as a method of predictive analysis of the oil and water content in pomace using portable or laboratory instruments. Therefore, the operation is not carried out in real time but requires at least 5–10 min to take the sample, place it correctly in the measuring cells of the instrument, and carry out the measurement. Considering the average mass feed rates of olive oil extraction plants, a result obtained after 5–10 min does not allow for the in line implementation of strategies to reduce oil losses in pomace. Furthermore, in large-scale mills that use many extraction lines, they would need to have an adequate number of measurements operators, and this would lead to a further increase in costs.

NIR spectroscopy is used in many food processes, such as for the in-line monitoring of (i) the temperature in sausages during industrial heat treatment, (ii) the performance of

membrane filtration in the whey protein fractionation process, (iii) the alcohol fermentation process in vinegar production, (iv) the moisture content during the apple chips process, (v) the quality parameters of deep-fried instant noodles, and (vi) food adulteration [36–39]. In the olive oil chain, NIR technology, albeit with offline tools, in addition to predicting water and oil content in pomace, is used in different applications, such as evaluating the olive ripening level, distinguishing between olive oil categories, and identifying herbicide residues in olives.

So far, no NIR technology has been developed to continuously analyze the characteristics of the pomace discharged from the decanter specifically designed to be implemented in line with the olive oil extraction plant.

The overall objective of the present study was the development and installation of a novel NIR device in an industrial olive oil extraction plant for in-line monitoring of the pomace discharged from the decanter. The development of a multivariate calibration model was executed for the real-time monitoring of moisture and oil content in pomace. The specific objectives of the present study were to evaluate the accuracy, robustness, and reliability of the spectral response obtained with the NIR sensor for monitoring the moisture and oil content of pomace during the extraction process, to identify the most important wavelengths with a multivariate data analysis, and, finally, to develop multivariate calibration models and apply the model to predict moisture and oil content in the pomace during the process.

2. Materials and Methods

2.1. NIR Spectroscopy Device

The FOSS company developed the device for this scientific study ProFoss^{TM} 2 (FOSS, Hilleroed, Denmark). The instrument operates in the NIR region from 850 to 1049 nm. The instrument uses high-resolution NIR diode array (DDA) technology, equipped with an external probe fiber with a circular geometry working with reflection technology to continuously monitor the process.

The most important specifications include (a) wavelength accuracy 0.5 nm, (b) wavelength precision < 0.02 nm, (c) wavelength stability < 0.01 nm/°C, (d) and the software package ISIscanTM Nova for instrument control.

The NIR device was connected via wi-fi with a personal computer that used FOSS ISIscan TM Nova Software Version 8.8.10 for the control, monitoring, and recording of the detected data.

2.2. Industrial Olive Oil Extraction Plant and NIR Device Implementation

The experimental tests were carried out at the Di Pietro Alfonso oil mill, Andria (BA). The plant (Figure 1) consisted of three parallel extraction lines fed by a single defoliator machine (model Gigante, Sivo Ettore Srl, Paolo del Colle, Italy). Each line involved a hammer crusher machine (model A60, Amenduni Nicola S.p.a., Bari, Italy), a malaxer group (model 3V6000, Amenduni Nicola S.p.a., Bari), a decanting machine (model REX 350, Amenduni Nicola S.p.a., Bari), and a disc separator (model A-3500, Amenduni Nicola S.p.a., Bari). The olive paste was moved by means of stator and piston pumps, while the wet pomace was moved by means of piston pumps. Each line had a mass flow rate of 6 t h^{-1}.

The transmittance interface probe of the NIR tool was installed directly on the outlet of the decanter, after the piston pump on the discharge pipe (Figures 1 and 2a,b).

2.3. Industrial Tests, Sampling, and Laboratory Analysis

The olive oil extraction experimental tests for the NIR device calibration were carried out from November 15th up to December 20th, 2023. During this period, the extraction plant worked regularly for 32 working days, processing olive fruits (*Olea europaea* L.) of the Coratina (60%), Leccino (20%), and Cima di Bitonto (20%) varieties. The olives were harvested mechanically and transported to the mill from 2 pm to 5 pm every day; then, they were processed on a first-come, first-served basis, usually within the next 10 h. The

malaxation time and temperature were, respectively, 60′ and 27 ± 1 °C. The decanter worked without adding process water.

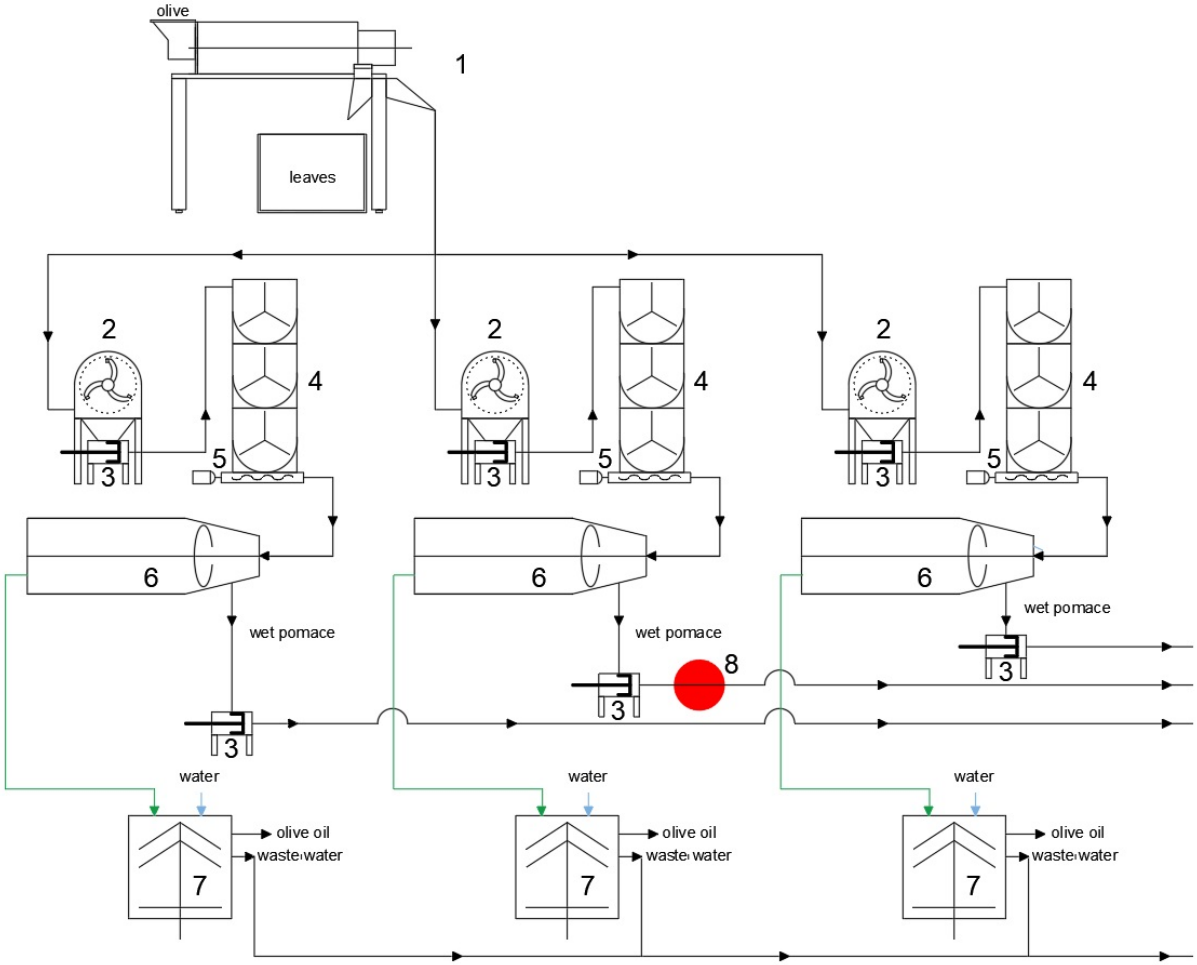
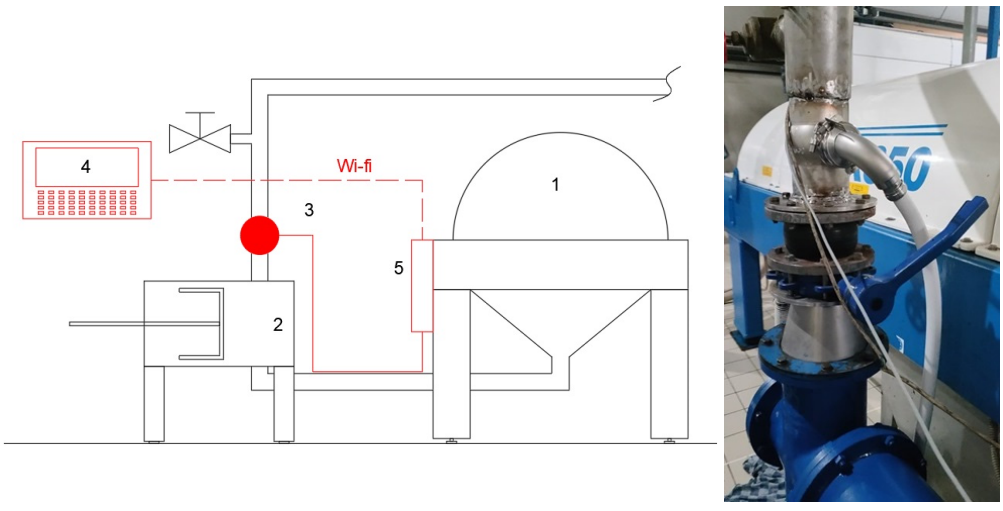


Figure 1. Layout of extraction plant: 1 defoliator, 2 hammer crushers, 3 piston pumps, 4 malaxing machines, 5 cavity pump stator, 6 horizontal centrifugal decanter, 7 vertical separator, 8 NIR probe.



(**a**) (**b**)

Figure 2. (**a**) Transmittance interface probe of the NIR tool scheme: 1 horizontal centrifugal decanter, 2 piston pumps, 3 NIR probe, 4 PC, 5 wi-fi transmitter. (**b**) Transmittance interface probe of the NIR tool photo.

For each of the 32 test days, at regular time intervals, 3 samples of pomace, weighing 0.5 kg, were collected immediately after the NIR probe, by the sample collection point equipped with a valve (Figure 2a). A total of 96 pomace samples were collected. For each sample, the exact time of sampling was recorded.

Moisture and oil content in the samples taken were then determined according to the official method [32].

2.4. Acquisition of NIR Spectra

During the olive oil extraction tests, the sample spectra were collected in the 850–1049 nm spectral range by taking an average spectrum of 64 scans, with a 0.5 nm resolution. The reference background spectrum was recorded using the internal reference of the NIR instrument. The spectra were collected continuously during the extraction process with an interval from each measurement of 3 s. At the same time, the moisture value and percentage of oil on a wet and dry basis were detected on the NIR device and recorded.

2.5. Sample Selection

Sample selection was conducted using Foss calibrator version 3.5 according to L.H. Xie et al. 2014. The multivariate approach based on the Global H (GH) was used to identify the anomalous spectra. To remove outliers, a threshold limit was set at the Mahalanobis distance (GH) of 3.0. By processing the 96 spectra of olive pomace collected, 9 anomalous spectra were eliminated. The remaining 87 spectra were used for development of calibration model and validation.

2.6. Spectra Pre-Processing

The calibration spectra obtained from the NIR device and analyzed with the reference method were employed to develop the calibration model. To remove variation in the spectra induced by light scattering, the absorption spectra were normalized by standard normal variate (SNV) [40]. This pre-treatment was applied to any value of absorbance at each wavelength, for all the individual spectra (Equation (1))

$$SNV_i = \frac{r_i - \bar{r}}{\sqrt{\frac{\sum_i^l (r_i - \bar{r})^2}{l-1}}} \quad (1)$$

SNV_i is the new value of absorbance obtained after pre-processing at the i-th wavelength. ri is the raw value of absorbance, $\bar{r}$ is the average of the absorbance values for the original spectrum, l is the number of wavelength values, and the denominator is the standard deviation of the spectrum.

2.7. Development of Calibration Models and Validation

The dataset obtained with the calibration samples was employed to develop PLS (partial least squares) models for moisture and oil content in pomace. These models were obtained by considering the overall spectral interval (850–1049 nm). The 87 spectra of pomace in the dataset were randomly split into a training set (80% of the dataset) and a test set (20% of the dataset).

The capability of the model to predict moisture and oil content in pomace was evaluated based on root mean square error of cross-calibration (RSMECV), root mean square error of prediction (RMSEP), and coefficient of determination (R^2) for calibration and prediction.

To evaluate the performance of the models, the BIAS and SLOPE parameters were also calculated.

Bias is the difference between the mean of one dataset and the mean of the other. Typically, bias is used to describe the difference between reference values and predicted values for a sample set. The value of bias is a measure of systematic errors occur during the calibration—when bias is 0, systematic errors are absent.

The slope of the model shows its symmetry: when a new model is calculated, it is normally perfectly symmetrical. The slope is 1, and the bias corresponds to the intercept, which is 0.

Considering that the outliers have a strong influence on the model's performance, these were identified and removed from the calibration set. In this study, outliers were identified and eliminated from the dataset when the difference between their actual and predicted values exceeded two times the RMSECV [39].

After outlier elimination, PCA was applied to the spectral data on both oil and water content in the pomace to obtain an overview of variation among samples. The data matrix was mean centered before PCA, and cumulative percent explained variance was used to choose the optimal number of principal components (PCs). The explained variance in a PC is the ratio (expressed as a percentage) between the variance in that PC and the total variance.

Multivariate analyses presented in this work were performed by using FOSS Calibrator version 3.5 by FOSS^{tm}.

3. Results and Discussion

3.1. Spectral Measurements and Investigation

The first step of the multivariate calibration was the use of NIR spectra detection to remove variation by SNV.

The profile of the collected spectra is presented in Figure 3a, while the spectra normalized by SNV are shown in Figure 3b.

The second step of the NIR spectra pre-processing was outlier detection. The outliers were preliminarily found by checking the spectra based on the Global H (GH).

As shown in Figure 3b, the spectrum in red, after the SNV, was found to be anomalous and was excluded from the dataset.

The GH value of the spectrum in red is 5.2, confirming an anomalous relationship between the variables, that could be due to an error in the preparation of the sample or in the collection of the measurement.

The trend was also investigated with the GH plot from the PCA model with four components obtained from spectra pre-treated with SNV (Figure 4).

On the basis of the Global H and PCA, the spectrum in red shown in Figure 3b was excluded from the dataset of the calibration model.

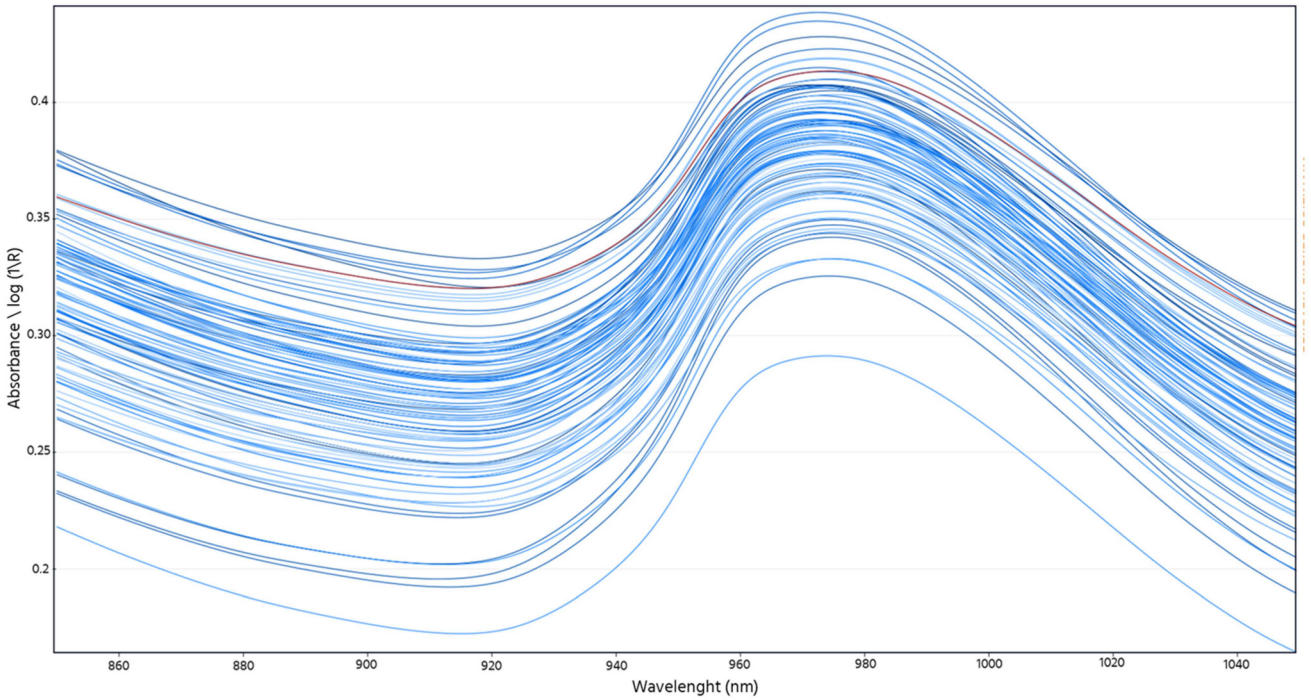
3.2. Calibration Results and Modelling

A dataset consisting in the calibration spectra of pomace was employed to develop a PLS model for moisture and oil content in pomace by considering the overall spectral range (850–1049 nm). The training set is formed by 77 spectra, whereas the test set consists of 18 spectra. The number of maximum components for building the model is set to 15. The optimal number of components is determined by considering the minimum value of the RMSECV; the cross-validation follows the scheme with four cancellation groups.

PLS Calibration of Pomace Oil and Moisture Content Model

The 87 samples used for the PLS model had minimum, maximum, and average values of moisture and oil content in the pomace, which are specified in Table 1 below.

The spectra of the training set and test set were mean centered before building the PLS model for oil content in pomace. The number of maximum components for building the model is set to 16. The cross-validation follows the scheme with four cancellation groups and the composition of the cancellation groups is randomly set. The results are shown in Figure 5.



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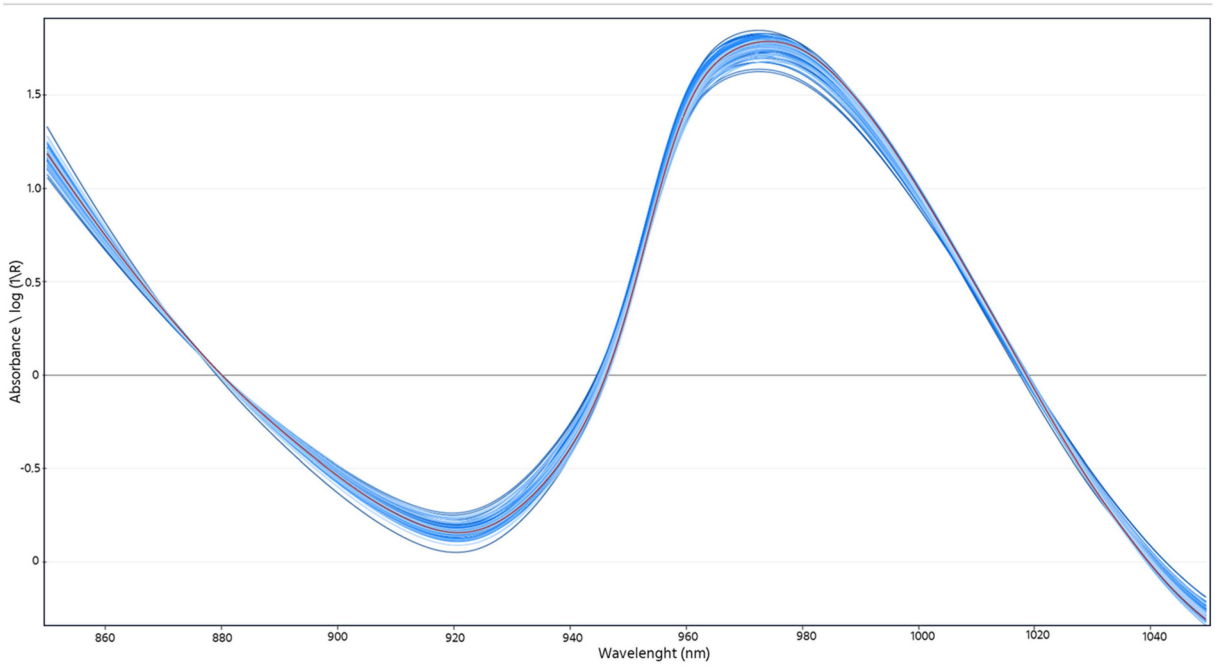

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Figure 3. NIR spectra of the dataset in the region 850–1049 nm: (**a**) raw spectra; (**b**) spectra normalized with SNV.

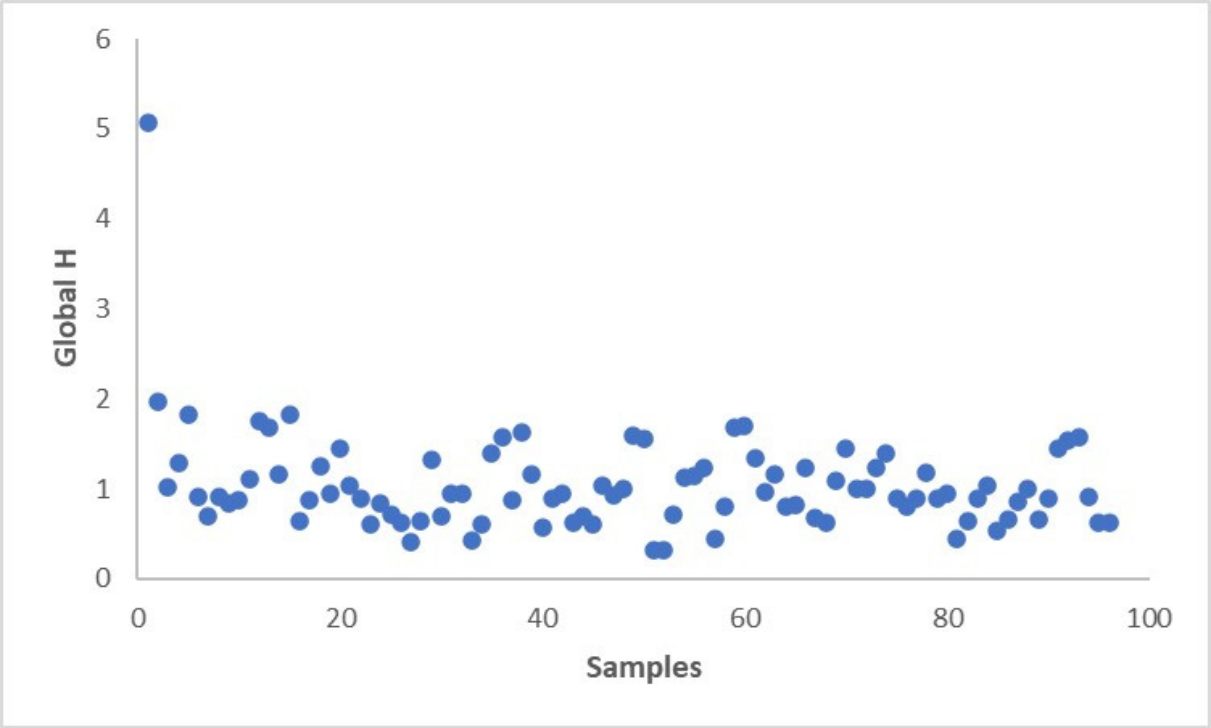


Figure 4. GH value for the NIR spectra of olive pomace.

Table 1. Moisture and oil content in pomace.

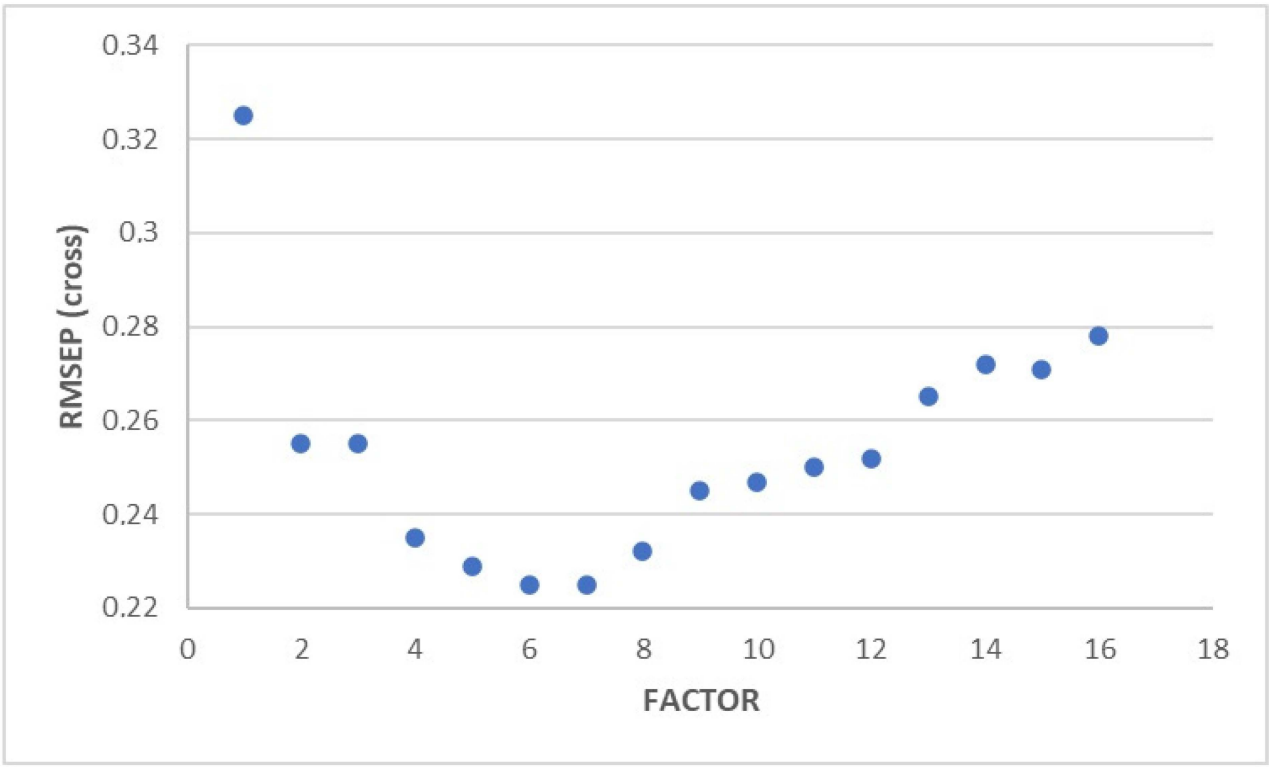
	Pomace Sample		
	Min	**Max**	**Average**
Oil content [%]	1.06	2.55	1.74
Moisture [%]	63.45	70.15	66.57

The optimal number of components for the PLS calibration of oil content is six. The spectra in red were identified and eliminated from the dataset considering that the difference between their actual and predicted values exceeded two times the RMSECV. The measured vs predicted results of the oil content in pomace after the removal of reference outliers are shown in Figure 6, and in Table 2, the calibration and validation results are summarized.

Table 2. Prediction results of the oil content in pomace for the calibration and validation model.

Calibration Performance					
Component	RMSECV	Bias	Slope	Intercept	R^2
Oil content	0.155	0.000	1.000	0.000	0.700
Validation performance					
Component	RMSEP	Bias	Slope	Intercept	R^2
Oil content	0.184	0.068	1.283	−0.406	0.773

A similar PLS model was built for humidity percentage in pomace, and the measured vs predicted results are shown in Figure 7. In Table 3, calibration and validation results are reported.



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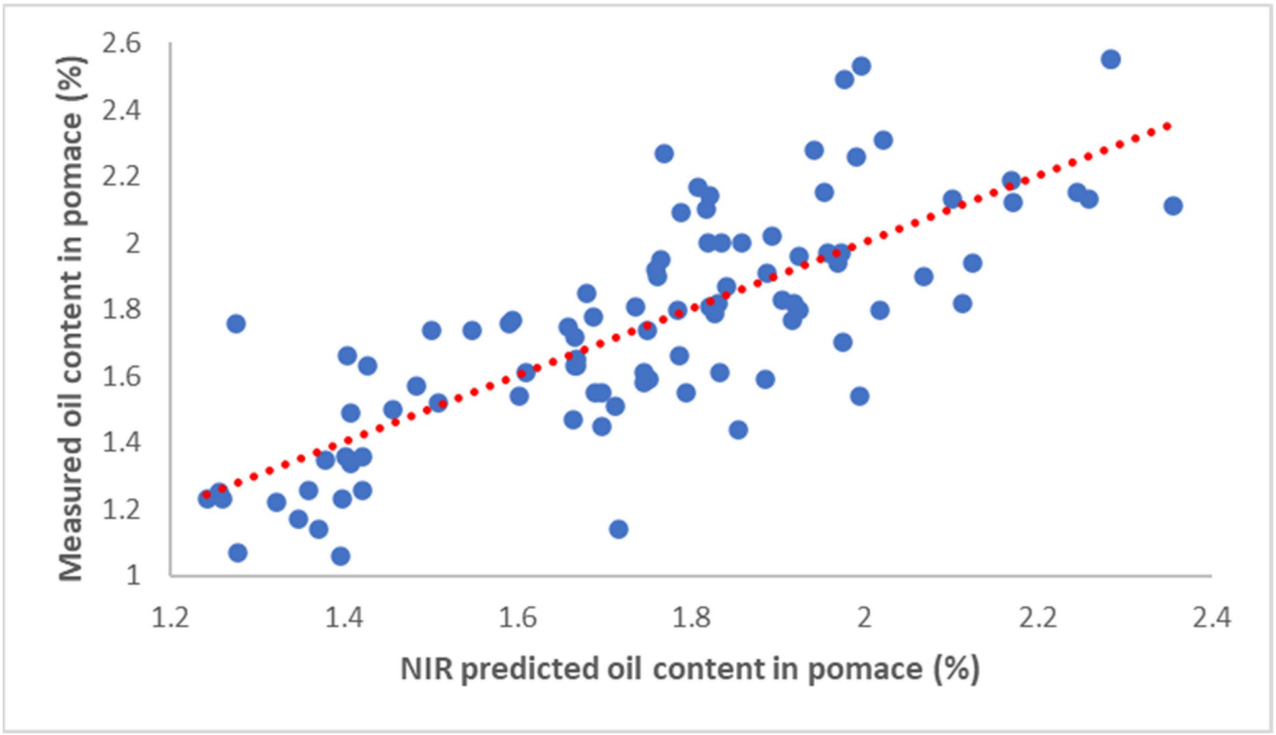

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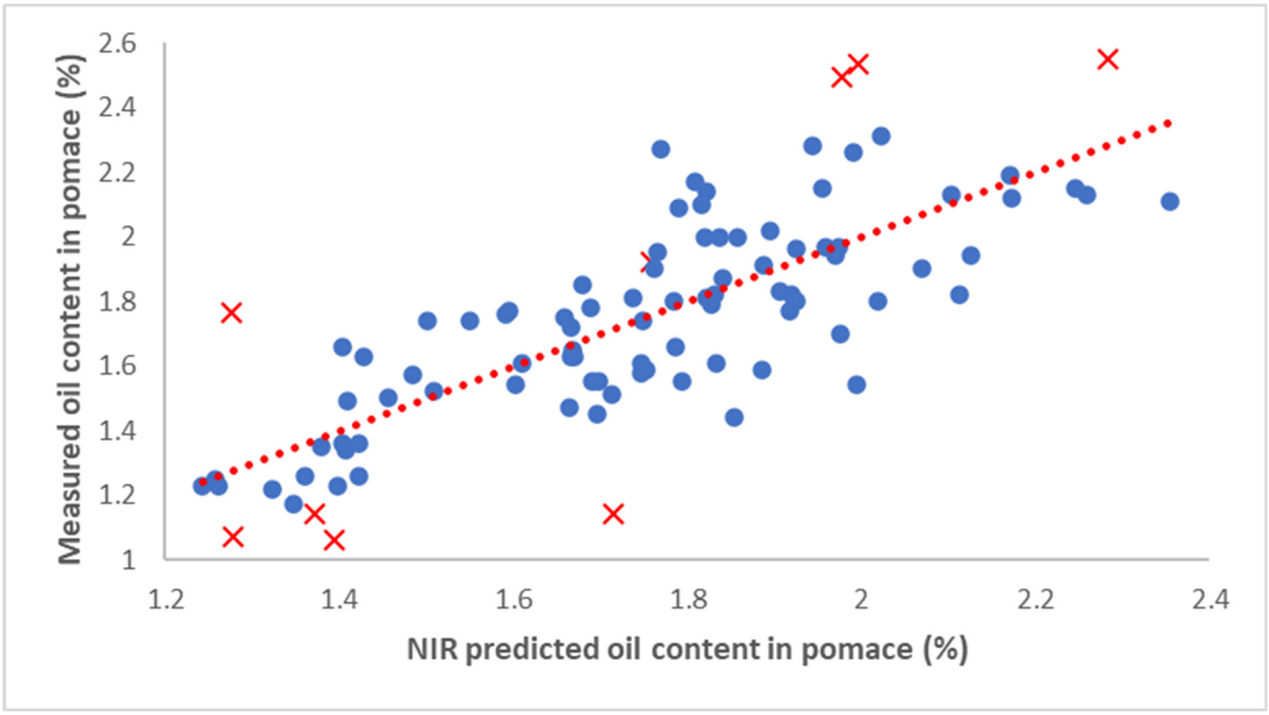
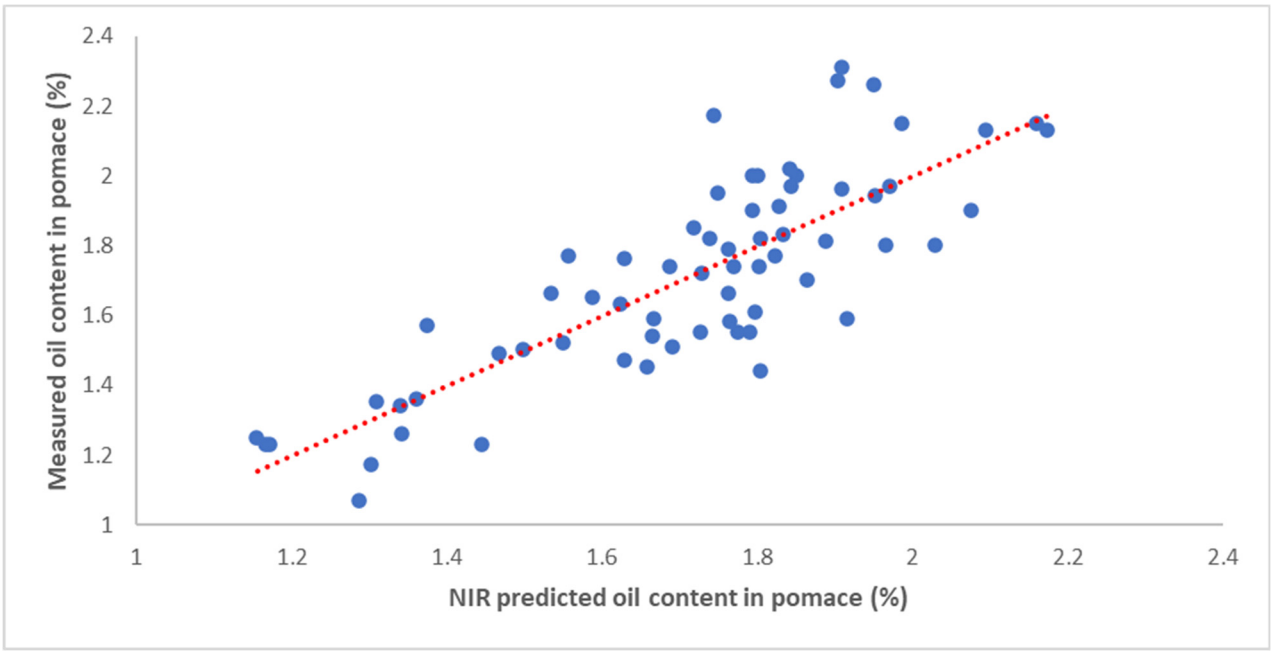
Figure 5. *Cont.*



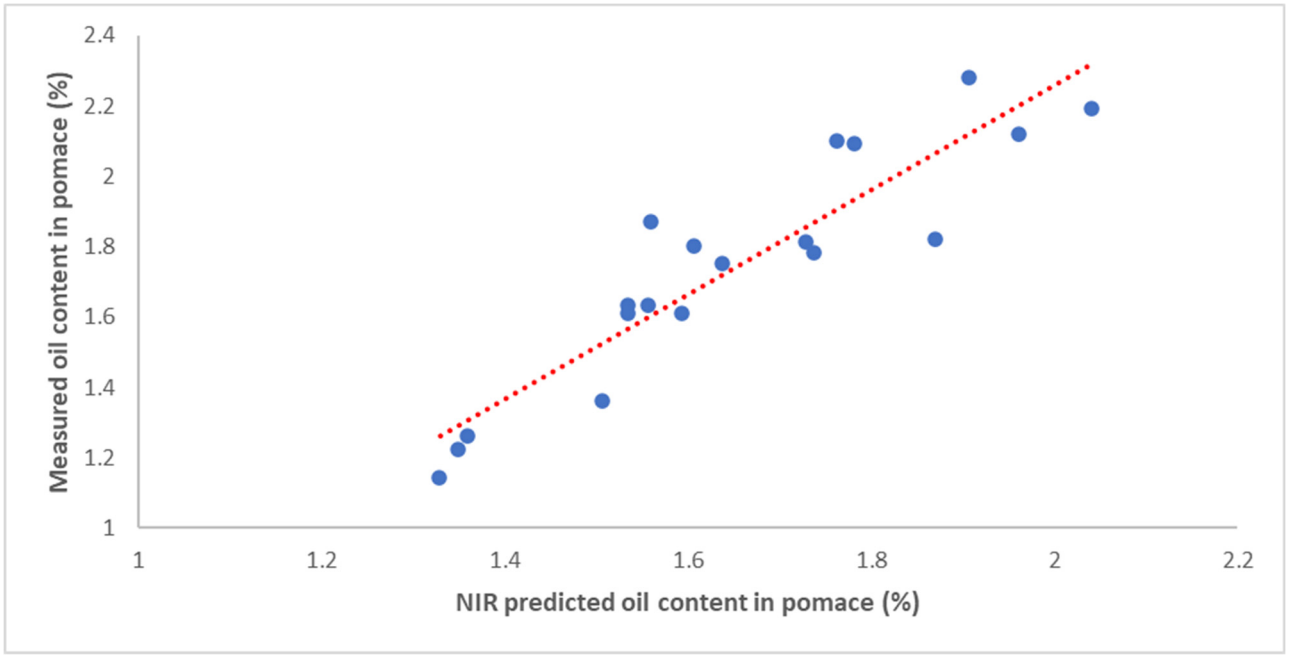
(**c**)

Figure 5. PLS cross-validation for oil content: (**a**) RMSECV vs number of components, (**b**) measured vs predicted values, (**c**) reference outliers Blue dots: measured vs predicted values, for each sample. Red cross: reference outlier sample. Dashed line: trend line of PLS model.



(**a**)

Figure 6. *Cont.*



(**b**)

Figure 6. Measured vs predicted values for oil content in the pomace: (**a**) calibration set; (**b**) validation set. Blue dots: measured vs predicted values for each sample. Dashed line: trend line of PLS model.

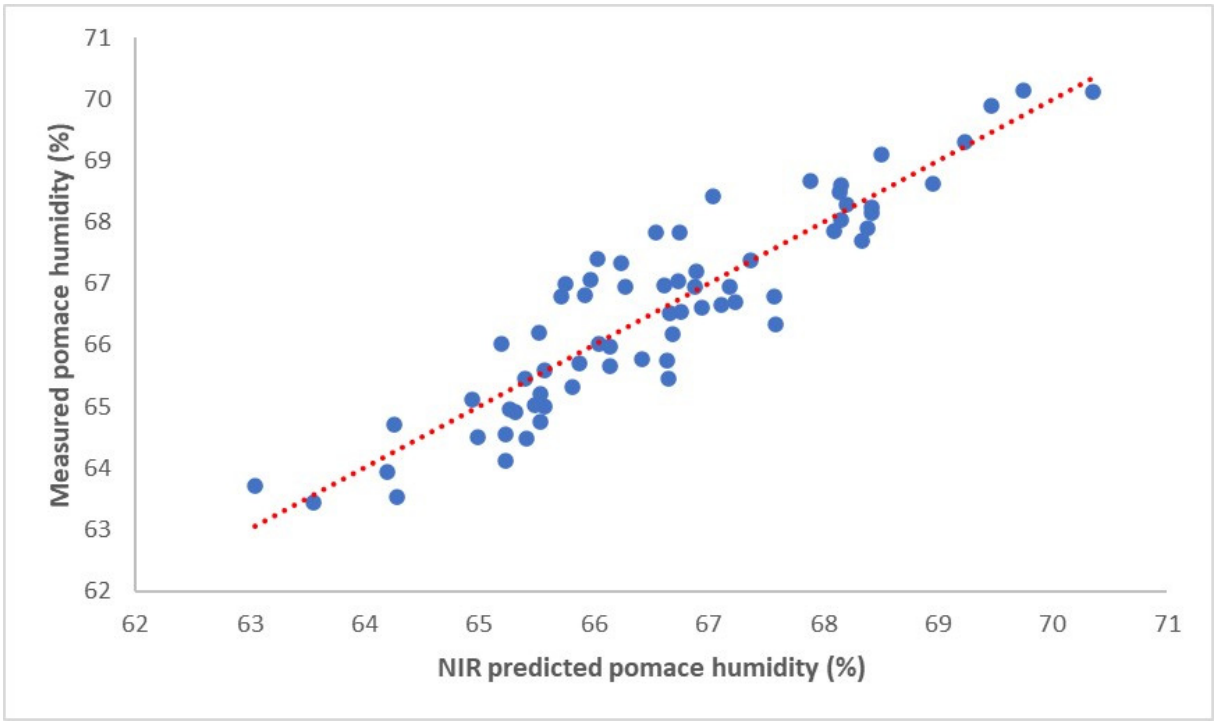
Table 3. Prediction results of the humidity percentage in pomace for the calibration and validation model.

Calibration Performance					
Component	RMSECV	Bias	Slope	Intercept	R^2
Humidity	0.653	0.000	1.000	0.000	0.829
Validation performance					
Component	RMSEP	Bias	Slope	Intercept	R^2
Humidity	0.698	0.066	0.894	7.110	0.676

Regarding the correlations of 0.700 and 0.829, these indicate a good relationship between the NIR spectra and oil and water contents, respectively, with a very low prediction error in both cases (0.155 and 0.653).

Considering the validation performance, the correlations of 0.773 and 0.676 for oil and water content, respectively, are certainly significant for in-line application in an industrial process, also considering the low prediction error values of 0.184 and 0.698 for oil and water content, respectively. The PLS models obtained on the spectra provide an accurate estimation of both parameters. In addition, bias values were very close to zero, for both models, thus identifying the absence of systematic variations. Bias is a measure of the systematic error in prediction and is calculated as the average, with the corresponding sign, of the residuals.

The prediction in terms of RMSEP both for oil and water content are coherent to the results obtained in previous research where NIR offline applications were used. Indeed, in both [33,34], using bench NIR spectroscopy operating in the 1100–2500 nm range resulted in an RMSEP of 0.21 and 1.80 for oil and water content in the pome fruit and 0.2 for oil and 0.8 for water content, respectively, for the two different studies carried out, just like the values obtained in the present study. In both studies, however, an R^2 value higher than 0.9 was obtained. The R^2 values are slightly higher compared to the value obtained in the present study, probably because the study was carried out with NIR spectroscopy operating within a wider range (1100–2500 nm) than that used in the present study (800–1100 nm).



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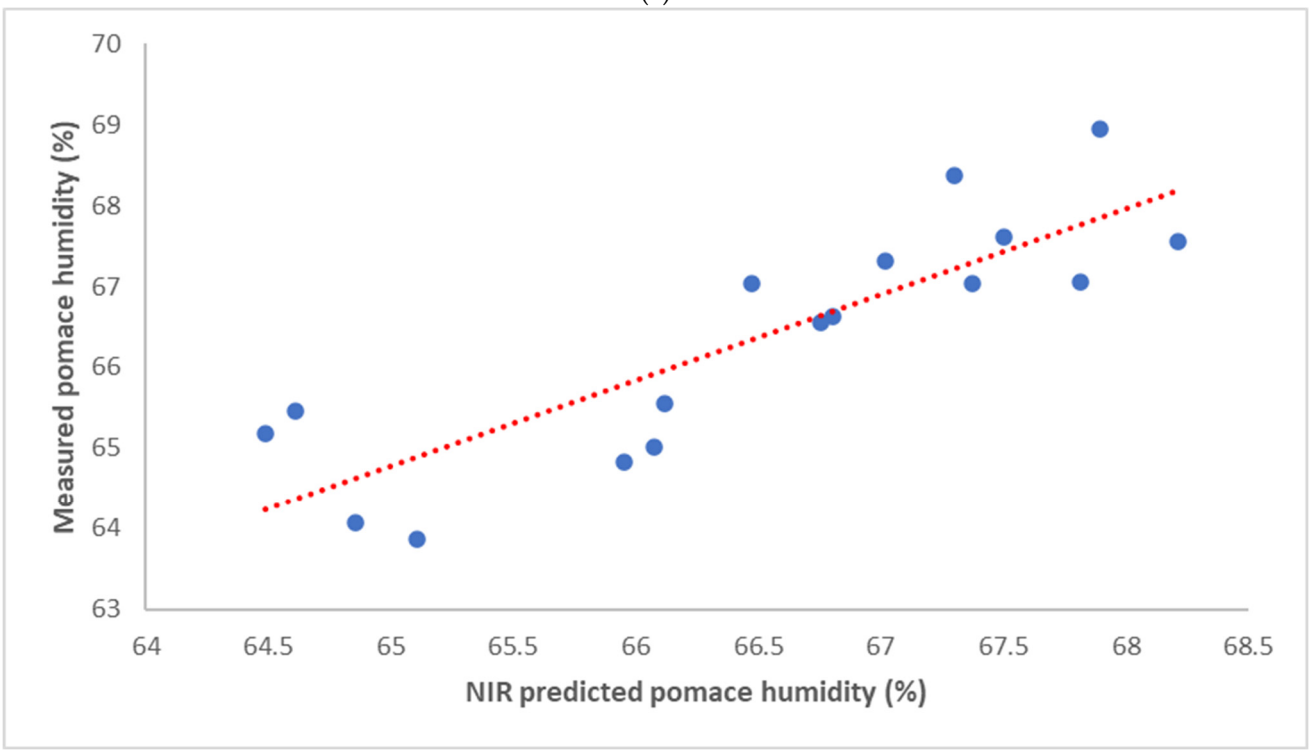

(**b**)

Figure 7. Measured vs predicted values for humidity in pomace: (**a**) calibration set; (**b**) validation set. Blue dots: measured vs predicted values for each sample. Dashed line: trend line of PLS model.

The results highlight the robustness of the NIR approach based on a PLS calibration model to monitor the industrial olive oil process.

4. Conclusions

The results derived from this scientific work have demonstrated that monitoring the percentage of moisture and fat in pomace produced at the end of the olive oil extraction process is effectively achievable by using in-line NIR setups on the olive oil process line implemented at the solid outlet of the decanter.

Despite some anomalous spectra being excluded from the database, the developed model demonstrated good and solid predictability in estimating the humidity of the pomace and the oil contained therein.

The results obtained can be interpreted as a first step towards the large-scale application of multispectral devices capable of rapidly determining the characteristics of the pomace leaving the extraction plant. In-line monitoring of pomace characteristics would give the plant manager a valuable tool to control the process to achieve the lowest level of oil loss in pomace during the process, and to efficiently learn how different plant/process settings lead to variations in the oil discharged from the decanter and in the percentage of oil extractability.

Our belief is that the integration of NIR tools and automatic plant setting systems combined with deep learning tools will actively contribute to increasing the efficiency of the extraction process, allowing for increased olive oil production with the same input raw materials. By increasing industrial extractability, it will be possible to avoid double oil extraction—which consists of virgin pomace reprocessing—with a consequent reduction in electricity and thermal energy and increase in the sustainability of the entire extraction process.

Further advances in the technology developed could concern the application of NIR technology in line for the evaluation of the olive oil and moisture of the olive paste fed to the decanter or for the evaluation of the oil lost in the waste products of extraction processes of other oily substances.

However, the ongoing and consistent efforts to enhance the NIR methodology represent an open-ended scope of research, also to other sectors not belonging to food processing, in order to achieve rapid and non-destructive analysis in real time. This will allow for quicker process intervention, improving efficiency.

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Conflicts of Interest: The authors declare no conflict of interest.

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