Simultaneous Concurrent Assessment of Extra Virgin Olive Oil Adulteration via Fourier Transform Mid-Infrared and UV-Visible Spectroscopy Combined with Partial Least Squares Regression

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Adulteration of olive oil is a common practice in the industry, where old and commercial oils are mixed with fresh olive oils. Adulteration can negatively affect the quality and authenticity of olive oil, leading to economic fraud and health concerns. Therefore, identifying and quantifying adulteration in olive oil is crucial for ensuring product quality and consumer protection. The objective of this study was to identify and measure the adulteration of fresh olive oils with old oil and commercial oil from the previous harvest year. The study aimed to achieve this goal using spectroscopic techniques in combination with chemometrics. Different spectroscopic techniques, such as FT-MIR and UV-vis spectroscopy, were utilized in this study. Partial least squares (PLS) regression was applied to predict the levels of adulteration in the samples with varying concentrations (0.84 - 52.13% w/w). Various pre-treatment methods were employed for both FT-MIR and UV-Vis spectral data. All the PLS models generated for FT-MIR and UV-Vis spectral data were successful in predicting the levels of adulteration, with high coefficients of determination for both calibration (0.963 - 0.995) and cross validation (0.935 - 0.993) models. The error values for calibration (0.621% - 2.728%) and cross validation (0.730% - 3.314%) were also low. Based on the results, it was found that the use of second derivative preprocessing for FT-MIR data and SNV preprocessing for UV-Vis data led to the best performance results in quantifying the level of adulteration of olive oil. Spectroscopic techniques in combination with chemometrics can be used to identify and measure the adulteration of olive oil.

Keywords: adulteration, olive oil, FT-MIR spectroscopy, UV-visible spectroscopy, PLS regression

Extra virgin olive oil (EVOO) is renowned for its exceptional quality, nutritional value, and unique sensory attributes [1-2]. However, the high demand and market value of EVOO have made it susceptible to adulteration and mislabeling, compromising consumer trust and product integrity. To combat this issue, the development of reliable and efficient methods for the simultaneous assessment of EVOO adulteration is crucial [3].

In recent years, Fourier transform mid-infrared (FT-MIR) and ultraviolet-visible (UV-Vis) spectroscopy, combined with chemometric techniques such as partial least squares regression (PLS), have emerged as powerful tools for food adulteration detection [4-6]. The synergistic application of these spectroscopic methods allows for a comprehensive and concurrent evaluation of EVOO authenticity and quality.

FT-MIR spectroscopy provides a molecular fingerprint of samples by measuring the absorption and transmission of infrared radiation in the mid-infrared range [7]. This technique enables the identification and quantification of various functional groups and chemical constituents present in EVOO [8]. UV-Vis spectroscopy, on the other hand, measures the absorption of light in the ultraviolet and visible range, allowing for the detection of chromophores and compounds responsible for color and absorbance characteristics [9].

By combining FT-MIR and UV-Vis spectroscopy with PLS regression, it becomes possible to develop robust calibration models that correlate the spectral information obtained from these techniques with reference values of EVOO composition, authenticity, and adulteration levels [10]. PLS regression, a multivariate statistical method, maximizes the correlation between the spectral data and the target variables, enabling the prediction of adulteration levels with high accuracy [11-13].

The simultaneous application of FT-MIR and UV-Vis spectroscopy with PLS regression holds several advantages for the assessment of EVOO adulteration. Firstly, it allows for rapid and non-destructive analysis, enabling real-time monitoring and quality control throughout the production and supply chain [14].

Secondly, the combination of spectral information from both techniques provides a more comprehensive and reliable assessment of adulteration, considering different aspects of EVOO composition and quality [15]. Lastly, the use of chemometric modeling ensures robust and accurate predictions, even in the presence of complex and overlapping spectral data [16].

In this article, we present a comprehensive study on the simultaneous and concurrent assessment of EVOO adulteration using FT-MIR and UV-Vis spectroscopy combined with PLS regression. We aim to investigate the effectiveness and practical applicability of this approach for the detection and quantification of adulterants commonly used in EVOO fraud, such as lower-grade olive oils and other vegetable oils. Additionally, we will explore the development of reliable calibration models and discuss the strengths, limitations, and future prospects of this integrated spectroscopic approach in the context of EVOO authentication and quality control.

Through this research, we strive to contribute to the development of robust analytical methods that can safeguard the authenticity, quality, and consumer trust in extra virgin olive oil, ensuring a transparent and reliable marketplace for this highly prized and valuable product.

Materials and Methods

Sample preparation

The samples used in this study were: One sample of extra virgin olive oil EVOO According to the analytical methods (EEC/2568/91) [17], recovered directly from an oil mill during the harvest season 2022 in December from the region of Beni mellal and was stored in dark glass bottles at refrigeration temperature (4 °C), and one sample of ordinary olive oil According to the analytical methods (EEC/2568/91), that was recovered accurately from an oil mill of the 2019 harvest season in the month of November from the region of Ouazzane and was stored in dark glass bottles at room temperature and away from the sun to prevent photo-oxidation. A sunflower oil was purchased from a local supermarket.

The EVOO sample used for the present analysis was adulterated with different samples of OOO and sunflower oil in a ternary mixture according to the relationship

$$\%Adulteration = \frac{(\mathbf{m}\,\text{sunflower oil} + \mathbf{m}\,\text{OOO})}{(\mathbf{m}\text{EVOO} + \mathbf{m}\,\text{sunflower oil} + \mathbf{m}\,\text{OOO})} * 100 \quad \text{(Eq. 1)}$$

$$m_{EVOO} + m_{sunflower\ oil} + m_{OOO} = m_{total}$$

Reagents and Materials

All solvents and reagents were of analytical or HPLC quality from Sigma-Aldrich. Acetic acid, Potassium hydroxide (KOH), Sodium hydroxide (NaOH), phenolphthalein, potassium iodide (KI), sodium thiosulfate ($Na_2S_2O_3$) were from Sigma-Aldrich (USA). Chloroform, cyclohexane, ethanol, Chloroform and diethyl ether were from Sigma-Aldrich HPLC grade (Steichein, Germany).

The masses of all components of the mixtures were measured using a 0.01 mg Mettler Toledo AG 245 analytical balance.

We used vortex to mix well the components of our ternary mixture.

Quality parameters

Four chemical parameters were tested before the acquisition of UV-visible and ATR-MIR spectra to ensure the categories of the olive oil samples used.

Acidity, peroxide value, UV indices and oxidative stability determinations According to the analytical methods (EEC/2568/91) described in the regulations of the European Commission we have determined the chemical indices of our olive oil samples such as Free Fatty Acids (FFA), expressed in percentage of oleic acid, the peroxide value (PV), expressed in meq O_2/kg of oil, and the specific extinction coefficients at 232 and 270 nm (K_{232} and K_{270}) [18].

Acidity test

Approximately 7 g of the dried, filtered, and well-mixed oils were weighed into a 250 mL Erlenmeyer flask. Then 50 mL of 96 % ethanol was taken and neutralized with 0.1 N aqueous sodium hydroxide (NaOH) solution in the presence of 2.0 mL of phenolphthalein solution to obtain a light, permanent pink color. Neutralized ethanol was added to the oil in the flask. The entire mixture was vigorously stirred and boiled on a hot plate for two minutes. Then, the contents of the flask were titrated with 0.1 N NaOH aqueous solution until a permanent, persistent pale pink color was obtained for one minute. The results can be expressed as percentage of acidity - free fatty acid (FFA) content, which means that the percentage of FFA expressed as a certain fatty acid of specified molecular weight depending on the type of oil under study. Normally, oleic acid with a molecular weight of 282 is used. In some cases, an average molecular weight, more appropriate to the nature of the oil, is used. It is calculated according to the following equation (Eq. 2) [19].

$$\text{Acidity \%} = (M \cdot V \cdot G \cdot 100)/(1000 \cdot W) \quad \text{(Eq. 2)}$$

Where:

M: Molarity of NaOH solution.

V: Volume of NaOH solution required to neutralize fatty acids present in the sample, mL.

G: Molar mass of oleic acid, in g/mol

W: Weight of the sample, g.

Peroxide value test

Approximately 5 g of oil was weighed into a 250 mL glass-closed conical flask. A solution of acetic acid and chloroform (3:1 V/V) was added (30 mL) with stirring to completely dissolve the oil. Saturated potassium iodide solution was added (1.0 mL). The flask was quickly closed and allowed to stand with occasional stirring for 1 min in the dark. Then 30 mL of freshly boiled and cooled water was added and about

0.5 ml of starch solution was added. The contents of the flask were titrated with 0.01 N sodium thiosulfate solution ($Na_2S_2O_3$) under vigorous stirring until the dark yellow or dark blue color had almost disappeared from the chloroform layer. A blank determination was performed in the same manner without using the oil. The peroxide value is the number of milliequivalents of peroxide found in 1000 g of the sample according to the following formula (Eq. 3) [20].

Peroxide value = $((V_1-V_2)\cdot M\cdot 100)/W$ (Eq. 3)

Where:

V_1: Volume of the thiosulfate solution used in test, mL.

V_2: Volume of the sodium thiosulfate used in blank test, mL.

M: Molarity of sodium thiosulfate solution.

W: Weight of the sample, g.

K-values test

The samples were analyzed using the VWR UV-1600PC spectrophotometer. A 0.25 g oil sample was weighed and placed in a 25 ml volumetric flask with cyclohexane solvent added. The samples were placed in a cuvette and their absorbance was measured at wavelengths between 232 nm and 276 nm. For cyclohexane, the maximum wavelengths were 270 nm and 232 nm. K_{232}, K_{270} were measured [21].

Spectral data and analysis

UV-Visible spectrometry. The absorbances of EVOO samples adulterated with OOO and sunflower oil in a ternary mixture were scanned from 200 to 800 nm by a VWR UV-1600PC UV-Vis Analyst spectrophotometer/software. The sampling interval was 2.0 nm with a fast scan rate, the quartz cuvettes were 1 cm long, and air was used as a blank.

Mid-infrared spectral acquisition. A JASCO FT/IR - 460 plus spectrometer was used to analyze the olive oil samples between 4000 and 600 cm^{-1} in absorbance mode with a spectral resolution of 4 cm^{-1}. Equipped with a deuterated triglycine sulfate detector (DTGS). A horizontal attenuated total reflectance (HATR) diamond accessory (Pike) was used for the sampling technique. A Globar source (MIR) and a KBr germanium separator, were used for recording FT-MIR spectra. Each sample was deposited on the HATR surface using a micropipette. After each analysis, the sample crystal was cleaned with acetone and deionized water. The spectra were processed with Spectrum Manager to remove the effect of H_2O and carbon dioxide and then transformed to dx format.

Chemometrics and preprocessing

Chemometrics it is a discipline of analytical chemistry that exploits statistical and computational tools to extract hidden information from laboratory data [22]. The data processed by chemometrics are often multivariate [23].

Partial Least Squares regression (PLSR) is a supervised regression method, widely used in Raman, Fluorescence and Infrared spectroscopy [24]. PLS is considered a chemometric multivariate calibration and prediction tool that identifies factors that maximize the covariance between two blocks of data. It finds factors (latent variables) among the observed variables X that explain the maximum variance of the variables C, applying the simultaneous decomposition of both. It discards unnecessary information from the regression, That is factors that account for large amounts of variance within the observed data and have zero correlation with the predictions [25].

All processes of processing UV-Vis (200-800 nm) and FT-MIR (4000-650 cm^{-1}) spectral data by applications of chemometric methods such as PLSR were performed using Unscrambler software, version 10.4 camo analytic.

Results and discussion

Physicochemical data analysis of olive oils

The physicochemical quality indices of the olive oils analyzed in this study, as shown in Table 1, reveal important insights. The first sample, obtained from the Beni-Mellal region, demonstrates values that align with the standards established by the International Olive Council (IOC) for the Extra Virgin Olive Oil (EVOO) category. Specifically, the acidity level is ≤0.8 %, the peroxide value is ≤20 meq O_2 kg^{-1}, and the UV absorption values, K_{232} and K_{270}, are ≤2.5 and ≤0.22, respectively. These results indicate that the olive oil from Beni-Mellal region meets the criteria for EVOO classification, reflecting its superior quality.

Table. 1. Physicochemical Analysis and Classification of Moroccan Olive Oils from Beni-Mellal and Ouazzane Regions.

Region	Year of harvest	Storage time (months)	Acidity (% oleic ac.)	IP (meq O_2/kg)	K_{232} (UV abs.)	K_{270} (UV abs.)	Category
IOC (2018)*	–	–	≤0.8	≤20.0	≤2.5	≤0.22	–
Beni-Mellal	2022	0	0.61	4.77	1.53	0.13	EVOO
Ouazzane	2019	36	2.23	9.84	2.10	0.18	OOO

Conversely, the second olive oil sample collected in the Ouazzane region during the 2019 harvest and stored for 36 months exhibits different characteristics. With an acidity level of 2.23 %, a peroxide value of 9.84 meq O_2 kg^{-1}, and UV absorption values of 2.10 (K_{232}) and 0.18 (K_{270}), it falls within the category of Ordinary Olive Oil (OOO). The extended storage period and higher acidity level suggest that this oil may have undergone some deterioration and no longer qualifies for the EVOO category.

FT-MIR spectra

This study analyzed 50 simulated FT-MIR spectra of adulterated extra virgin olive oils, examining the entire spectral range of 4000-600 cm^{-1}. The HATR-MIR spectral data in Figure 1 revealed distinct bands corresponding to the functional groups present in the olive oil. Within the spectral region of 3000-2800 cm^{-1}, two intense bands at 2920 cm^{-1} and 2852 cm^{-1} represented the symmetrical and asymmetrical elongation vibrations of the CH_2 and CH_3 groups. Deformation vibrations of these groups were observed at 1463 cm^{-1} and 1377 cm^{-1}. The elongation vibrations of the C=O group of the lipids appeared around 1743 cm^{-1}. In the spectral zone of 1200-900 cm^{-1}, characteristic bands associated with sugars were present. Significant phenol bands were identified in the 722 cm^{-1} region. These findings provide valuable insights into the distinctive spectral features and functional groups present in the FT-MIR spectra of the adulterated extra virgin olive oils.

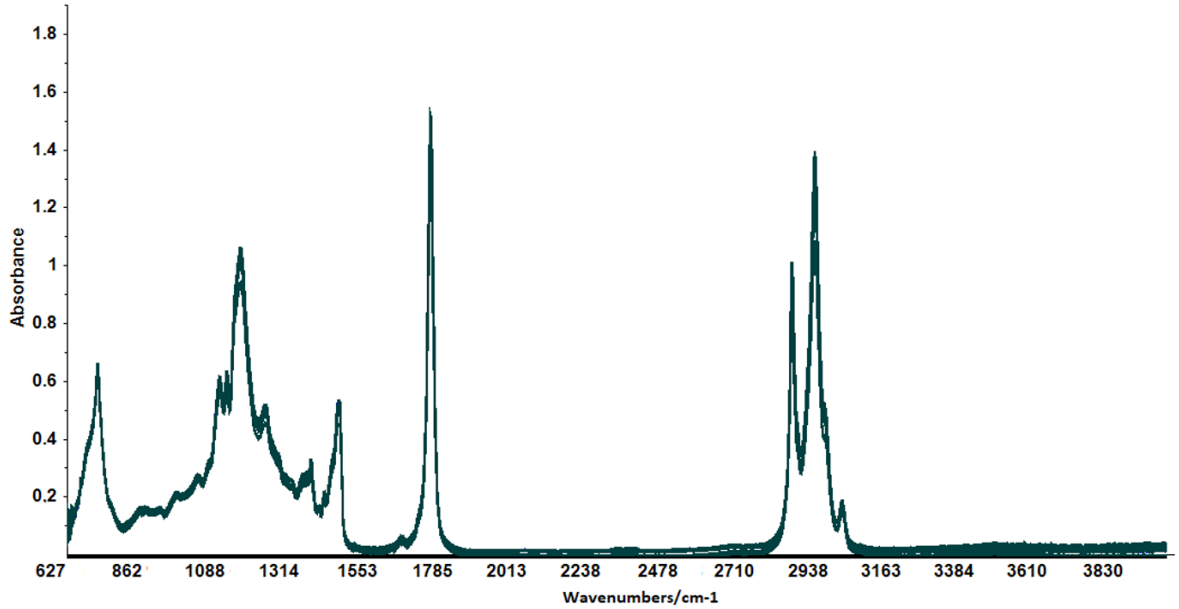

Fig. 1. FT-MIR spectra of 50 olive oil samples.

Table. 2. FT-MIR absorption frequencies and their functional group attributions in the 4000-600 cm^{-1} region [17].

Frequencies	Attributions
3006 cm^{-1}	Trans=C-H stretching
2920 and 2852 cm^{-1}	Symmetric and asymmetric stretching of -CH_2
1743 cm^{-1}	Stretching of -C=O
1463 cm^{-1}	Bending of -CH_2
1377 cm^{-1}	Bending of -CH_3
1237 cm^{-1}	Stretching of -C-O
1161 cm^{-1}	-C-O stretching; -CH_2 bending
1118 cm^{-1}	-C-O stretching
1096 cm^{-1}	-C-O stretching
722 cm^{-1}	Cis-CH=CH- out-of-plane bending

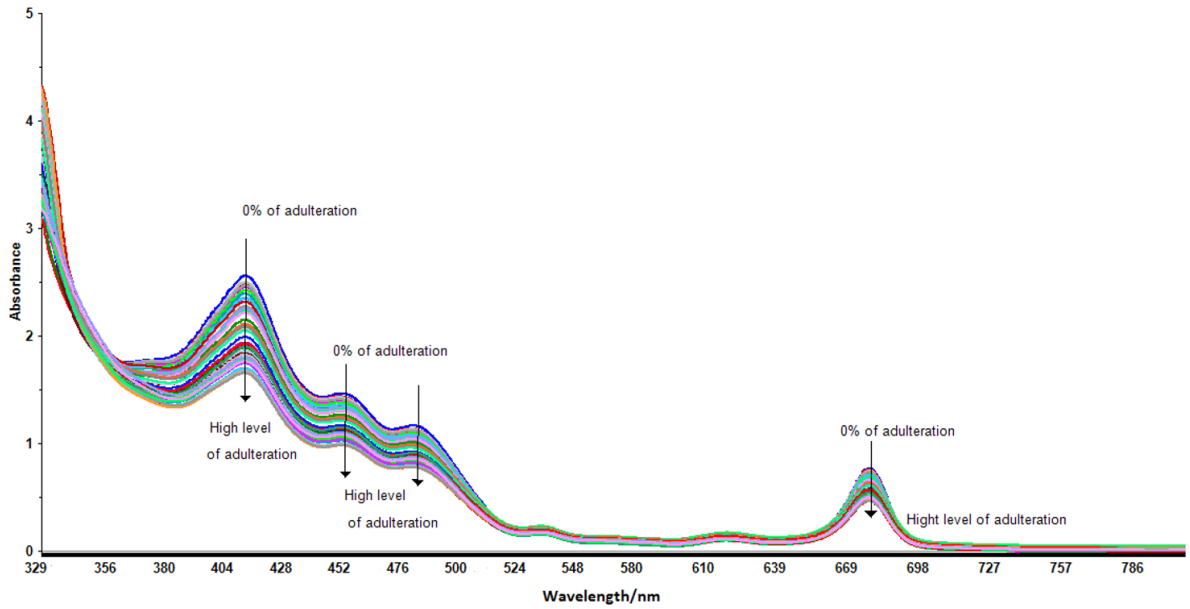

Fig. 2. UV–Vis spectra of 50 olive oil samples.

UV–Vis spectra

The UV-visible spectra of the olive oil samples are presented in Figure 2. These spectra exhibit specific peaks between 230-270 nm, indicating the presence of conjugated dienes and trienes of unsaturated fatty acids. Furthermore, the 300-400 nm band is associated with various polyphenols [26]. Differences in the quality, variety, and geographical origin of the olive oil samples, as well as measurement parameters, may explain any shifts in peak positions or the absence of certain peaks in the current study compared to previous findings. Additionally, carotenoids, which are responsible for color, exhibit absorption between 430-460 nm, while the peak at 670 nm is attributed to chlorophylls and their derivatives [26].

Figure 2 demonstrates that the primary differences in the UV spectra of the samples are noticeable in the peaks attributed to carotenoids (400-500 nm) and chlorophylls (670 nm). Both carotenoids and chlorophylls are pigments that can be influenced by environmental factors such as light and temperature, and can be transformed or degraded during storage. As a result, any discrepancies observed in the UV spectra of samples that have been stored for extended periods may be associated with alterations in their pigment composition. The oxidation of these compounds during storage is thought to contribute to changes in pigment composition [27].

Prediction of adulteration levels

In this study, FT-MIR and UV-Visible spectroscopy and partial least squares (PLS) regression were used to quantify the adulteration of olive oil. A total of 50 adulterated samples were collected, with varying levels of adulteration.

The results of actual versus predicted percentages of olive oil adulteration (0.84-52.13% w/w) determined by FT-MIR spectroscopy are presented in Fidures 3,4.

The results of PLSR for different preprocessing techniques of FT-MIR (EVOO adulterated with old oil (OOO) and sunflower oil (SO)) are summured in Table 3. As can be seen in Table 3, the PLS regression model was able to accurately predict the level of adulteration in the validation set with an R-squared value of 0.971 - 0.988. The root-mean-square error of calibration (RMSEC) was between 2.167 - 0.618% indicating that the model can predict the level of adulteration with high accuracy for the three type of adulterant.

Cross-validation is an important tool for evaluating the performance of multivariate models, such as PLS regression. In this study, the CV method was used to evaluate the performance of all PLS models for the quantification of adulteration in olive oil. The results demonstrated that the all PLS models developed using FT-MIR spectra were able to accurately predict the adulteration level of olive oil with an R^2 of 0.982, 0.948, and 0.975 for T, OOO and SO respectively. These results demonstrate that both raw and preprocessed data provide accurate results and that the highest result was observed in preprocessing the data by second derivatization. These results also show that the R^2 and RMSE of the calibration and cross-validation are closely matched, demonstrating the lack of overfitting.

The UV-visible results demonstrate that PLS regression gives good results for the quantification of olive oil adulteration by a binary mixture. This performance is explained by the high value of R^2, which varies between 0.963-0.995 and the low value of RMSEC that varies between 0.624 % - 2.728 % as can be seen in Table 4 and Figure 4.

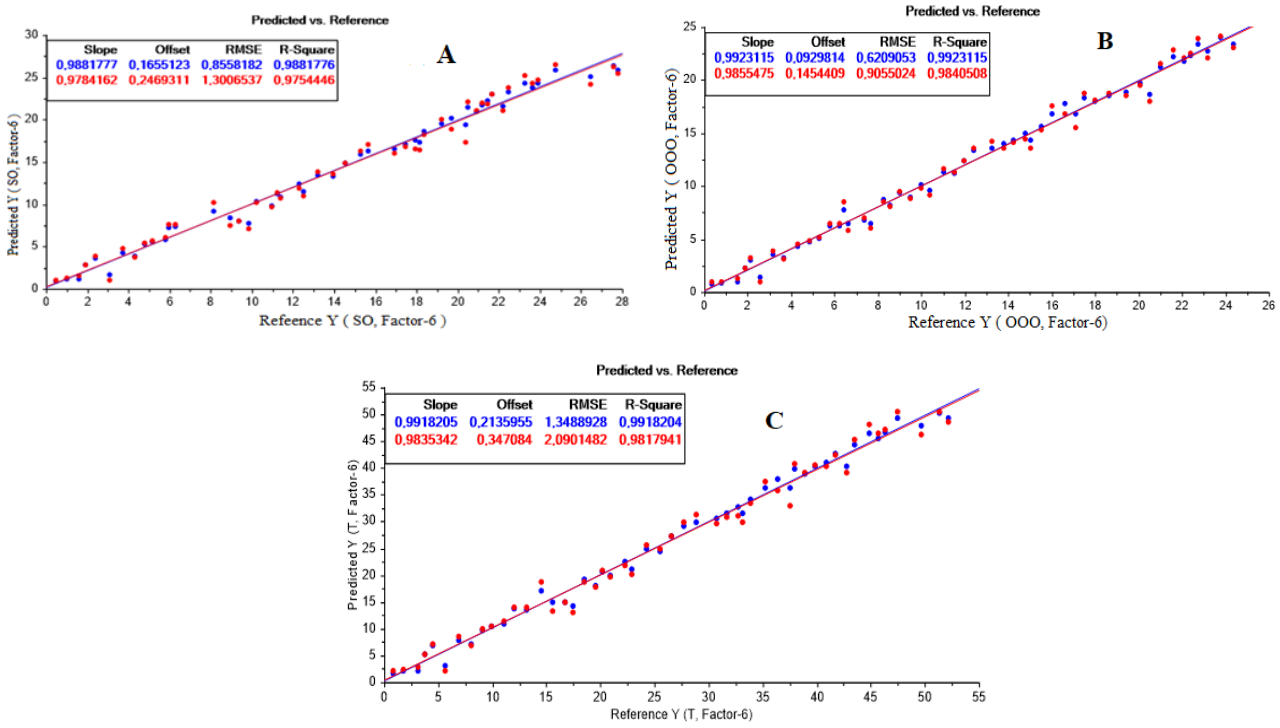

Fig. 3. Actual versus predicted percentages of olive oil adulteration (0.84-52.13% w/w) determined by FT-MIR spectroscopy (A: Sunflower oil (SO), B : Ordinary olive oil (OOO), and C: Total adulteration in EVOO (SO + OOO) = T), (Blue: calibration, Red: cross validation).

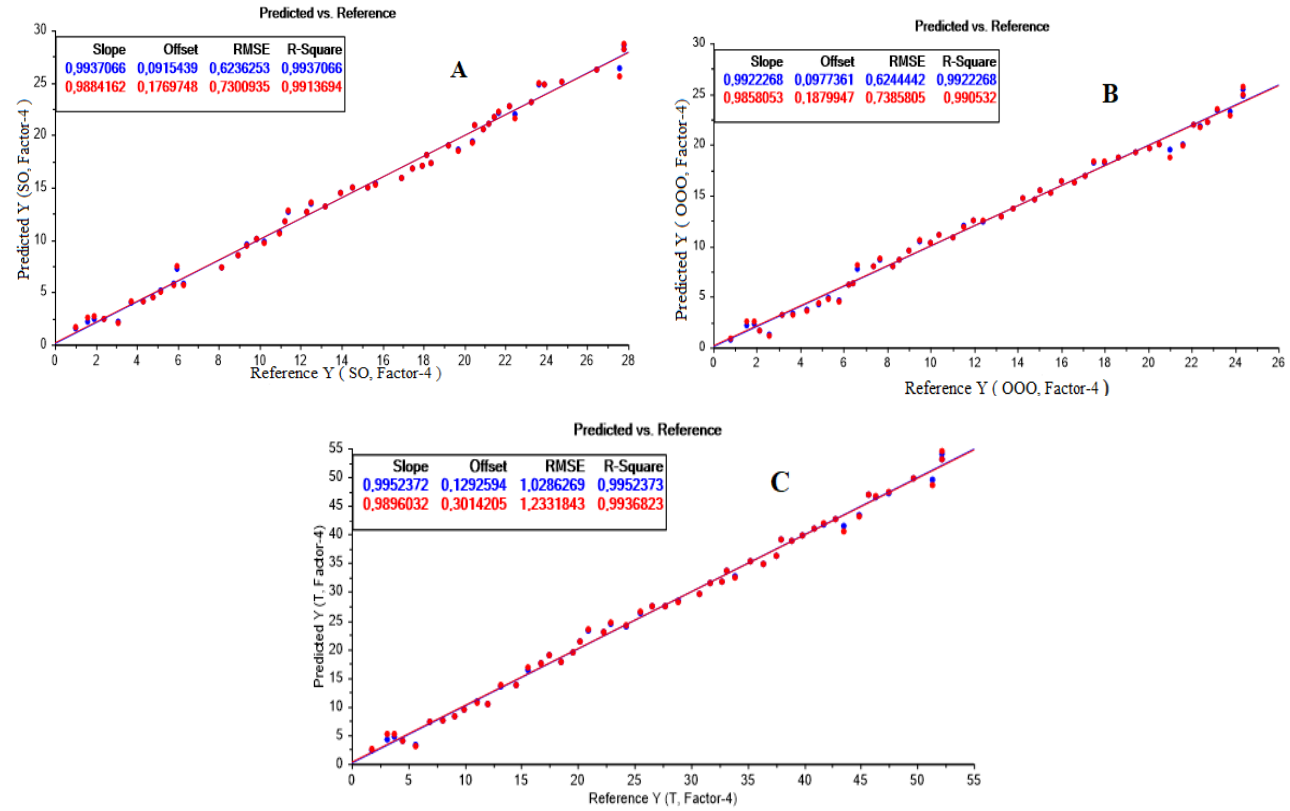

Fig. 4. (A, B, C) Actual versus predicted percentages of olive oil adulteration (0.84-52.13 % w/w) determined by UV-Vis spectroscopy (Blue: calibration, Red: cross validation).

Table. 3. The results of PLSR for different preprocessing techniques of FT-MIR (EVOO adulterated with old oil (OOO) and sunflower oil (SO)).

Preprocessing	Type	R^2cal	RMSEC	R^2cv	RMSECV
Without preprocessing	SO	0.973	1.299	0.935	2.026
	OOO	0.983	0.954	0.954	1.525
	T	0.979	2.167	0.946	3.473
Detrend polynomial degree 1	SO	0.987	0.882	0.961	1.507
	OOO	0.993	0.618	0.978	1.011
	T	0.991	1.396	0.972	2.414
Detrend polynomial degree 2	SO	0.988	0.856	0.975	1.300
	OOO	0.992	0.621	0.948	0.905
	T	0.992	1.348	0.982	2.090
SNV	SO	0.971	1.333	0.935	1.994
	OOO	0.984	0.902	0.961	1.414
	T	0.979	2.124	0.951	3.314

Table. 4. The results of PLSR for different preprocessing techniques of UV-Vis (EVOO adulterated with old oil (OOO) and sunflower oil (SO)).

Preprocessing	Type	R^2cal	RMSEC	R^2cv	RMSECV
Without preprocessing	SO	0.993	0.644	0.988	0.798
	OOO	0.989	0.732	0.983	0.916
	T	0.993	1.182	0.988	1.540
Detrend polynomial degree 1	SO	0.963	0.644	0.990	0.801
	OOO	0.989	0.732	0.984	0.910
	T	0.993	1.182	0.990	1.524
Detrend polynomial degree 2	SO	0.963	1.500	0.954	1.701
	OOO	0.965	1.306	0.957	1.492
	T	0.966	2.728	0.957	3.108
SNV	SO	0.993	0.632	0.991	0.730
	OOO	0.992	0.624	0.990	0.738
	T	0.995	1.028	0.993	1.233

Evaluation of these regression models by cross-validation shows good results for all models built on data pre-transformed by different spectral processing methods, and that the highest R^2 values and the lowest RMSECV values are obtained using the SNV method.

The use of cross-validation also shows that 4 principal components are sufficient to build a powerful model for the quantification of adulteration without a lack of fit as the performance parameters obtained by the calculation are very close to those of cross-validation as shown in Figure 4.

In the case of FTIR analysis, the PLS method identified six latent variables, also known as factors, as the most informative components for building the prediction model. These six latent variables represent underlying patterns or structures in the FTIR data. They capture the variations in the spectral features that are most relevant to explaining the variations in the adulteration level. By using these six latent variables, we were able to construct a robust prediction model that accurately relates the FTIR measurements to the outcome variable of interest.

On the other hand, for the UV-Vis analysis, the PLS method revealed that only four latent variables were necessary to construct a reliable PLS model. These four latent variables represent the most significant spectral information(X) that contributes to explaining the variance in adulteration level (Y). While the UV-Vis technique measures different aspects of the sample compared to FTIR, the PLS analysis still identified relevant features in the UV-Vis spectra, which enabled us to build an effective prediction model with a reduced set of factors.

The difference in the number of selected latent variables between FT-MIR (six) and UV-Vis (four) suggests that the two spectroscopic techniques

capture different aspects of the underlying relationship between X and Y. The FT-MIR data appears to have more complex underlying structures, as indicated by the need for a larger number of latent variables to adequately represent the variation. In contrast, the UV-Vis data, though informative, seems to have a simpler underlying pattern, and therefore, a smaller number of latent variables is sufficient to construct a reliable prediction model.

Overall, these findings highlight the suitability of both FT-MIR and UV-Vis techniques for predicting the outcome variable Y, albeit with differing degrees of complexity. Understanding these differences can aid researchers and practitioners in selecting the most appropriate spectroscopic method based on the nature of their specific research or application.

In our pursuit to develop a robust olive oil adulteration prediction model, the pivotal insights derived from the Partial Least Squares Regression (PLS-R) loadings plot, as depicted in Figure 5, have proven instrumental. Within this plot, we discerned two primary regions of significance, unveiling the critical factors underpinning the accuracy of our predictive model. The foremost region, spanning from 1700 cm^{-1}, corresponds to the fingerprint region within the mid-infrared spectrum, revealing distinctive peaks associated with ν(C=O) and C–H stretching bands. These spectral signatures play a pivotal role in the accurate detection of olive oil adulteration. Furthermore, our investigation extended to the UV-Vis domain, where the loading plot exhibited two prominent contributions at 415 nm and 680 nm. These findings underscore the multifaceted nature of our olive oil adulteration prediction model, with distinct spectral regions offering invaluable insights into the detection and prevention of adulteration in this precious commodity.

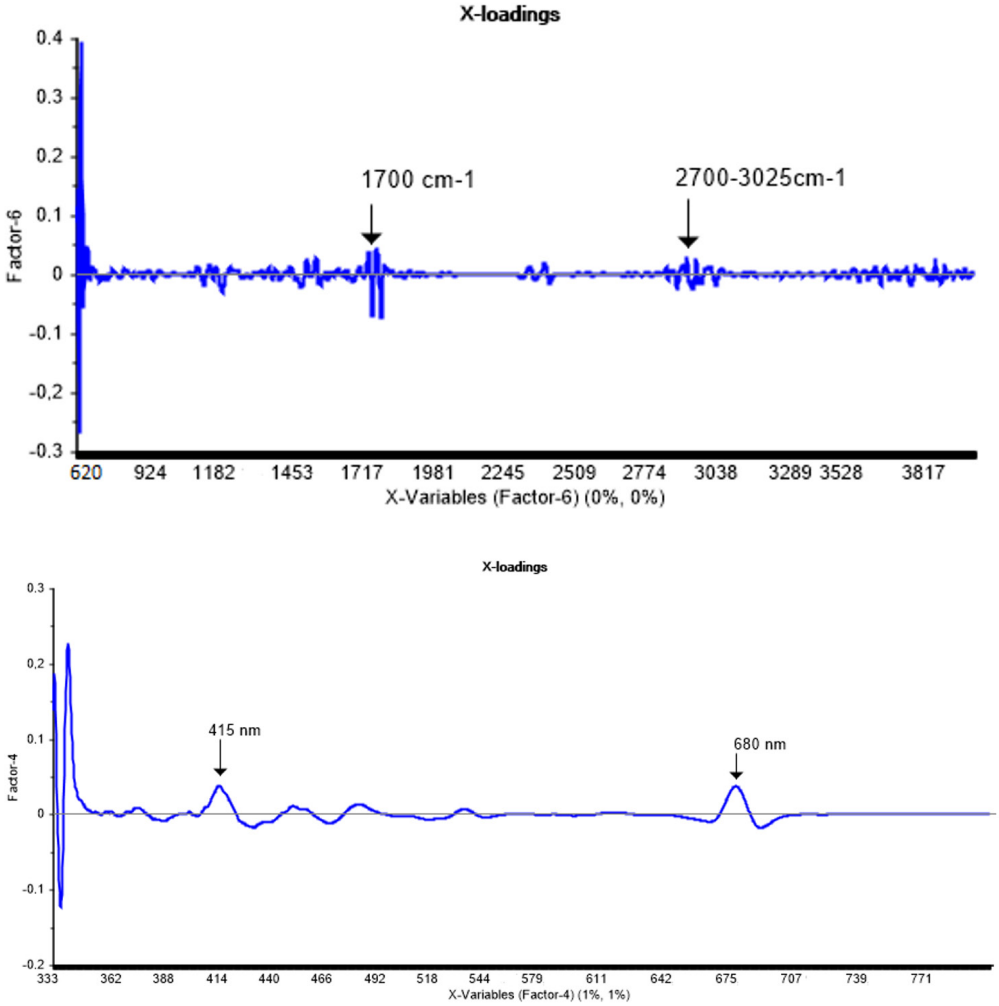

Fig. 5. Loading plot of PLSR of FT-MIR and UV-Vis spectroscopy*.

General discussion

The performance of the two methods, FT-MIR and UV-Vis, was evaluated using different preprocessing techniques for the analysis of EVOO adulterated with Ordinary Olive Oil (OOO) and sunflower oil (SO).

For the FT-MIR technique (Technique 1), the results show that without any preprocessing, both OOO and SO types achieved high R^2cal values, indicating good calibration performance. However, the RMSEC values were relatively higher, suggesting larger errors in the predicted values. The R^2cv and RMSECV values indicated good cross-validation performance, but the RMSECV values were higher compared to the calibration errors. Applying detrend polynomial degree 1 and 2 as preprocessing techniques improved the FT-MIR results. The R^2cal values increased, indicating better calibration performance, and the RMSEC values decreased, indicating reduced errors in prediction. The R^2cv and RMSECV values also improved, demonstrating enhanced cross-validation performance. Among the preprocessing techniques, detrend polynomial degree 2 consistently yielded higher R^2cal and R^2cv values and lower RMSEC and RMSECV values, suggesting better predictive performance overall. Using the SNV preprocessing technique for FT-MIR resulted in slightly lower R^2cal and R^2cv values compared to the other techniques. The RMSEC and RMSECV values were relatively higher, indicating slightly larger errors in prediction. However, the differences in performance between SNV and the other preprocessing techniques were relatively small.

Moving on to the UV-Vis technique (Technique 2), without any preprocessing, both OOO and SO types achieved high R^2cal and R^2cv values, indicating excellent calibration and cross-validation performance. The RMSEC and RMSECV values were relatively lower, suggesting smaller errors in prediction compared to FT-MIR without preprocessing. Applying detrend polynomial degree 1 as a preprocessing technique improved the UV-Vis results. The R^2cal and R^2cv values remained high, indicating good calibration and cross-validation performance, while the RMSEC and RMSECV values decreased, indicating improved predictive accuracy. However, when using detrend polynomial degree 2 as a preprocessing technique, the UV-Vis results showed decreased performance compared to the other techniques. The R^2cal and R^2cv values were lower, indicating reduced calibration and cross-validation performance, and the RMSEC and RMSECV values increased, suggesting larger errors in prediction. Applying the SNV preprocessing technique for UV-Vis resulted in consistently high R^2cal and R^2cv values, indicating excellent calibration and cross-validation performance. The RMSEC and RMSECV values were relatively lower, suggesting smaller errors in prediction.

In summary, both FT-MIR and UV-Vis techniques demonstrated good performance for the analysis of EVOO adulterated with OOO and SO. The choice of preprocessing technique had a notable impact on the results. For FT-MIR, detrend polynomial degree 2 yielded the best performance, while for UV-Vis, both no preprocessing and SNV techniques showed favorable results. Selecting the appropriate technique would depend on the specific requirements and objectives of the analysis.

Conclusion

Based on the results, it can be concluded that the combination of FT-MIR and UV-Vis spectroscopy with PLS regression can be an effective method for the quantification of olive oil adulteration. Both spectroscopic techniques offer complementary information, with FTIR providing information about the functional groups present in the sample and UV-Vis providing information about the absorbance of specific compounds. PLS regression allows for the development of a model that can accurately predict the level of adulteration based on the spectral data.

This study has shown that the combination of FT-MIR and UV-Vis spectroscopy with PLS regression can accurately detect and quantify the adulteration of olive oil with lower-quality oils or other vegetable oils. The method has also been shown to be sensitive to low levels of adulteration, making it a useful tool for quality control in the olive oil industry.

Overall, the use of FT-MIR and UV-Vis spectroscopy combined with PLS regression offers a rapid, non-destructive, and reliable method for the quantification of olive oil adulteration. However, it is important to note that the accuracy of the method is dependent on the quality and variability of the spectral data used to develop the PLS model.

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Conflicts of Interest

All authors declare no conflict of interest.

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