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Detection of the Presence of Hazelnut Oil in Olive Oil by FT-Raman and FT-MIR Spectroscopy

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The detection of the presence of refined hazelnut oil in refined olive oil at low percentages is still a challenge with the current official standards. FT-Raman and FT-MIR spectroscopies have been used to determine the level of detection of the presence of hazelnut oil in olive oil. Spectroscopic analysis has been made not only with the entire oil but also with its unsaponifiable matter. Univariate and multivariate statistical models have been designed with this objective. This study shows that a complete discrimination between olive and hazelnut oils is possible and that adulteration can be detected if the presence of hazelnut oil in olive oil is >8% and if the blends are of Turkish olive and hazelnut oils. The limit of detection is higher when the blends are of edible oils from diverse geographical origins.

KEYWORDS: Olive oil; hazelnut oil; FT-Raman; FT-MIR; chemometrics

INTRODUCTION

The nonexistence of official analytical methods regarding the detection of olive oil adulteration with hazelnut oils and the fact that consumers are very sensitive to adulterations are the two main motivations for several services of the European Commission and international institutions (i.e., IOOC) involved in the prevention and detection of fraud in the olive oil sector. The adulteration of olive oil with other edible oils can be of three ways: blends of virgin oils, blends of refined oils, and blends of virgin and refined oils. Nowadays, one of the most concerning adulterations is carried out with hazelnut oils (*Corylus avellana* L.). Recently it has been reported that quantities of hazelnut oil are being imported into the European Community, without proper declaration to Customs and Excise, and it is suspected that it is being used to adulterate olive oils bottled within the Community. In the case of this adulteration, the addition of refined hazelnut oil to virgin olive oil can be detected, even at very low percentages of the adulterant, by the standard method based on quantification of stigmastadienes (*1*). However, there is no standard that detects the presence of cold-pressed hazelnut oil in virgin olive oil, if the resulting blend does not smell of hazelnut, or the presence of refined hazelnut oil in refined olive oil or olive oil (blend of refined and virgin olive oils) at percentages lower than 20%. The first kind of theoretical adulteration does not seem to be lucrative due to the low odor threshold of the filbertone, the hazelnut oil marker. The alternative would be to carry out the blend with lampante olive oil that might mask the hazelnut oil odor (*2*). However, the lampante olive oil must be refined prior to being sold to consumers, which means that the problem is, in fact, only one: the detection of the presence of refined hazelnut oil in refined olive oil.

Numerous methodologies have been proposed, although most of them are focused on the detection of the presence of crude hazelnut in virgin olive oil. A great part of these methodologies quantifies chemical compounds that are removed during the refining process. Thus, several analytical methods have been proposed for the detection of filbertone [(*E*)-5-methylhept-2-en-4-one] (*3*), which is a hazelnut oil marker (*4*), whereas other researchers have proposed to determine chlorophylls (*5*), tocopherols (*6*), and phenols (*7*). The real challenge, however, is the detection of refined hazelnut oil in olive oil as the triglycerides and the compounds of the unsaponifiable matter of these edible refined oils are very similar (*8*). The research in this field has been focused on the quantification of fatty acids, triglycerides, and sterols by chromatography or spectroscopy. Chromatography has been used to distinguish hazelnut oil from olive oil on the basis of the triglyceride (*9*) or sterol (free and esterified) (*9*, *10*) profiles. A mathematical program, based on the differences between theoretical and empirical triacylglycerol compositions, has also been used to distinguish the presence of refined hazelnut oil at low percentage (*11*).

Some chromatographic methods have been successful (*12*), but they are time-consuming and require access to laboratory facilities. An alternative is spectroscopy, which has not been

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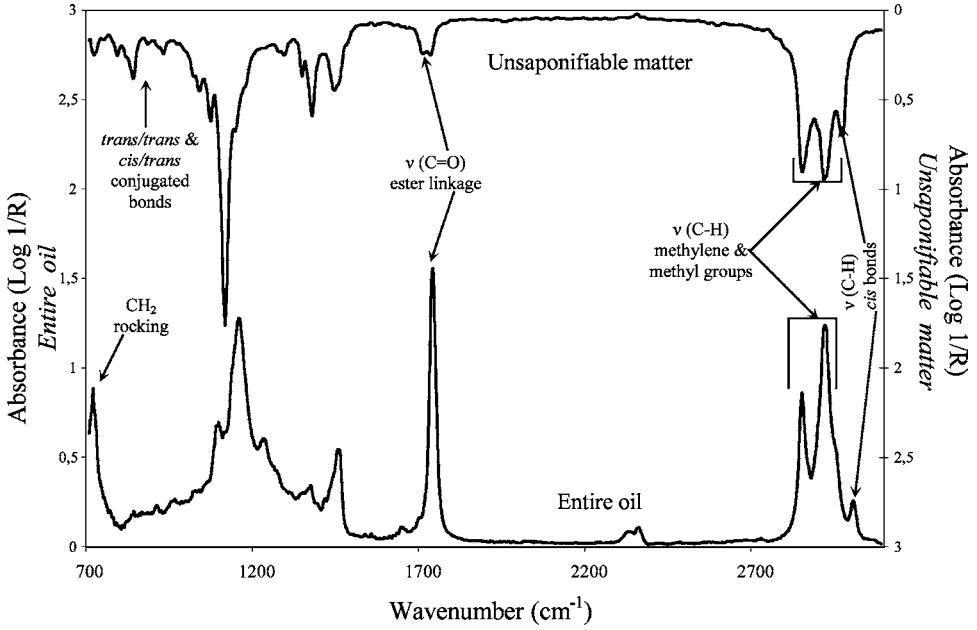

Figure 1. FT-MIR spectra of an entire oil and its unsaponifiable matter (reverse scale).

widely applied to the detection of this kind of adulteration. Proton NMR has been used alone (*13*) or combined with ^{31}P NMR (*14*) to distinguish genuine hazelnut oils from genuine olive oils; the merging of the information from ^{13}C NMR and ^{1}H NMR, with the assistance of mathematical algorithms, has been able to detect the presence of the refined hazelnut oil at percentages lower than 10% (*15*).

FT-Raman and FT-MIR vibrational spectroscopy have also been demonstrated to be successful (*16*), and their approaches are faster and simpler than the previous techniques. From a chemical point of view, both Raman and infrared spectroscopies are based on the vibrational transitions occurring in the ground electronic state of the molecules. Raman scattering arises from the changes in the polarizability or shape of the electron distribution in the molecule as it vibrates, whereas infrared absorption requires a change of the intrinsic dipole moment with the molecular vibration (*16*). Previous studies have stated not only the undoubted characterization of several edible oils by FT-Raman and FT-MIR spectroscopy (*17*) but also the techniques that might be used for detecting the adulteration of olive oil with other vegetal oils even with similar fatty acid profiles (*18*−*24*), later checked by other authors (*25*−*27*).

This work shows the results of using FT-Raman and FT-MIR spectroscopy to detect the presence of hazelnut oil in olive oil at low percentages. A large set of samples, from several geographical origins, were analyzed in the course of a European research project (*28*). The study was performed on the entire oil and on its unsaponifiable matter. Stepwise linear discriminant analysis (SLDA) was applied to extract, interpret, and exploit the information from the spectra.

MATERIALS AND METHODS

Samples. FT-Raman and FT-MIR techniques were applied to the set of samples produced within the MEDEO European project (*28*). The number of samples of the training set was 189, and 44 samples were analyzed in the test set (blind samples). The admixtures of the training samples were prepared with refined and lampante virgin olive oils from several geographical origins (Greece, Italy, Morocco, Spain, Tunisia, and Turkey) and two kinds of hazelnut oils (refined and crude) from France, Italy, Spain, and Turkey. The percentage of hazelnut oil in olive oil varied from 2 to 20% (2, 5, 8, 11, 14, 15, 17, and 20%). The preparation of the training samples was carried as follows: ∼50% of the samples were prepared by mixing Turkish olive oils with Turkish hazelnut oils, ∼20% of the samples were prepared with oils from other geographical origins, and 30% of the samples were genuine olive oils. The selection of the test (blind) samples was based on four assumptions: (i) the current major problem is the adulteration of refined olive oils with refined hazelnut oils; (ii) the most common blends are carried out by adding Turkish hazelnut oils to Turkish olive oils because the cheapest hazelnut oil is produced in Turkey (its production equals 80% of hazelnut world production); and (iii) there are always possibilities of fraudulent mixtures whatever the variety or the geographical origin of the edible oils. Raman and MIR spectra were collected not only from the entire oil but also from the unsaponifiable matter extracted from each oil (*29*). All of the spectroscopic analyses were carried out in duplicate.

FT-MIR Analysis. The FT-MIR spectra (4000−900 cm^{-1}) were acquired with an AEGYS MI2000 XS FT-IR spectrometer from Pertstorp Anadis (Anadis Instruments USA, Inc.). This instrument is equipped with a Michelson interferometer and an ATR crystal. A horizontal ATR crystal (ZnSe crystal with six internal reflections) plate was used to collect the spectral data of oil and unsaponifiable samples. Prior to the analysis, the ATR crystal was cleaned with hexane and wiped dry before putting the sample on its surface. For the analysis of the unsaponifiable matter the samples were dissolved in hexane before being placed on the crystal, and the dissolvent was evaporated before the spectral analysis. The reference spectrum used was the air spectrum collected before each sample analysis, the resolution was set at 4 cm^{-1}, and the number of scans collected for each spectrum was 50.

FT-Raman Analysis. FT-Raman spectra were acquired on a Perkin-Elmer (Boston, MA) NIR-FT-Raman spectrophotometer 2000R equipped with a Nd:YAG laser source emitting at 1064 nm (9394 cm^{-1}). The 180° backscattering refractive geometry and an InGaAs detector have been used. The spectrometer was managed through the Spectrum for Windows software of Perkin-Elmer. The spectral data were obtained with a resolution of 4 cm^{-1} and a nominal laser power of 600 mW. For each spectrum, 50 scans were co-added and averaged to get a good signal-to-noise ratio. The unsaponifiable matter has been diluted to 25% (w/w) with carbon tetrachloride and introduced in NMR tube series 500 from Sigma-Aldrich with an internal diameter of 5 mm and a length of 75 mm (Bornem, Belgium) before Raman analysis. The oil fraction of the samples has been introduced in classical test tubes having an internal diameter of 12 mm and a length of 75 mm and kept in a water bath at the temperature of 40 °C prior to analysis. All of the Raman analyses were performed by using a thermostated sample holder designed to maintain the sample at a constant temperature of 45 °C.

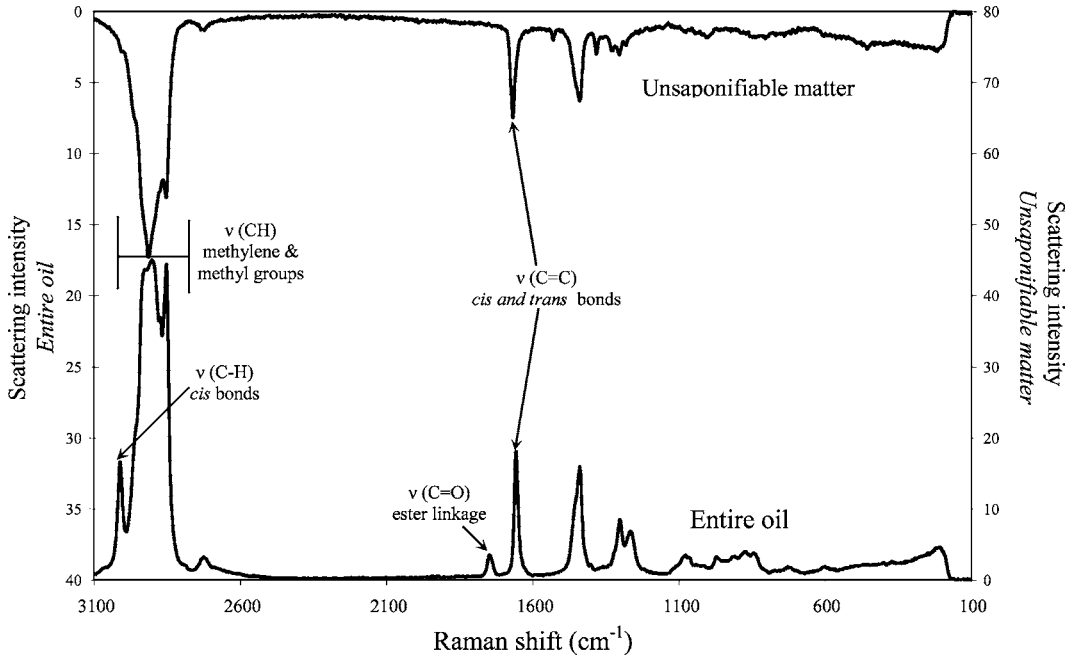

Figure 2. FT-Raman spectra of an entire olive oil and its unsaponifiable matter (reverse scale).

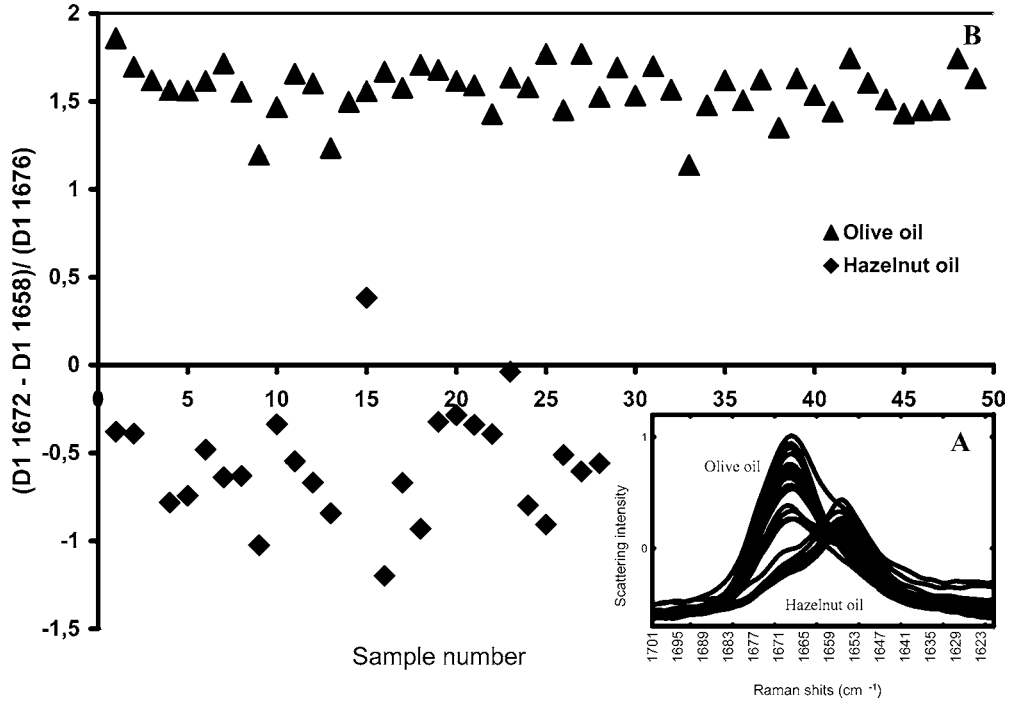

Figure 3. FT-Raman spectra of the unsaponifiable matter of hazelnut and olive oils (**A**) and classification of the samples obtained with the rule based on three Raman shifts (1676 1672, and 1658 cm^{-1}) (**B**).

The Savitsky−Golay eight-point smoothing function (*30*) was applied to all of the spectra, and then a first-derivative mathematical treatment was performed over 15 convolution points. Univariate statistics was used to carry out the repeatability study; the relative standard deviation was lower than 12%. The supervised multivariate procedure of stepwise linear discriminant analysis was used to select the Raman shifts of the mathematical models under the strictest conditions (*31*). Chemometric analyses were performed using Excel v. 7.0 (Microsoft Corp., Redmond, WA) and Statistica v. 6.0 (Statsoft, Tulsa, OK) (*32*).

RESULTS AND DISCUSSION

Figures 1 and **2** show, respectively, the FT-MIR and FT-Raman spectra of the oil and the unsaponifiable matter of an olive oil sample.

A visual analysis of the spectral databases (entire oil and unsaponifiable matter) was first performed. The FT-Raman spectra of the pure olive oils (OO) and pure hazelnut oils (HAZ) reveal that the absorbance or the scattering intensity at one frequency did not allow visual discrimination between the two groups. However, the unsaponifiable spectra of OO and HAZ samples showed clear differences between the groups.

Figure 3A shows part of the FT-Raman spectra (1701−1621 cm^{-1}) of pure OO and HAZ samples where the most important differences between the two groups were observed by the Fisher coefficient (*33*). This region is characteristic of a stretching vibration of C═C from specific compounds present in the samples. More precisely, the bands centered around 1670 and

Table 1. Summary of the Results of the SLDA Models Constructed Using the Training Samples

spectroscopy/kind of sample	variables in the model	selected wavenumbers (cm^{-1})	percentage of correct classification		
			OO[a]	HAZ[a]	mixtures
FT-Raman/entire oil	10	1276, 1282, 1309, 1659, 1693, 1670, 1674, 2948, 2981, 3001	85.7	100	86.7
FT-Raman/unsaponifiable	7	1669, 2966, 2977, 2983, 2994, 3004, 3006	95.0	100	97.5
FT-MIR/entire oil	10	717, 1080, 1088, 1099, 1111, 1122, 1126, 1296, 1439, 1450	90.5	94.4	83.3
FT-MIR/unsaponifiable	8	714, 980, 987, 1439, 1446, 1628, 1631, 1728	95.5	100	100

[a] OO, olive oil; HAZ, hazelnut oil.

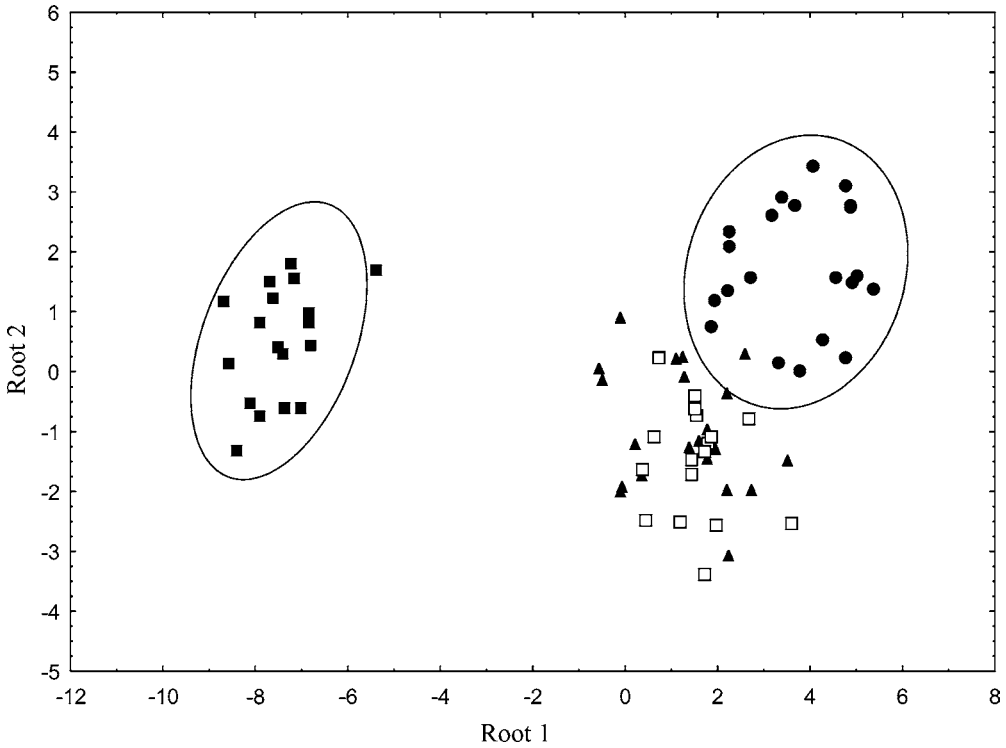

Figure 4. FT-MIR spectra of the unsaponifiable matter of the test samples: classification of genuine olive oil (●) and hazelnut oil (■) and their mixtures [>10% (□) and <10% (▲)] by SLDA. Genuine edible oils are delimited by ellipses of confidence at $\alpha = 0.90$.

1660 cm^{-1} are, respectively, characteristic of trans and cis unsaturated groups. In the unsaponifiable FT-Raman spectra, the band centered near 1670 cm^{-1} has been mainly attributed to the presence of squalene. This is the major trans-polyunsaturated hydrocarbon of the olive oil unsaponifiable matter. Using the scattering intensities measured at three different Raman shifts (i.e., 1672, 1658, and 1676 cm^{-1}), a decision rule (R1) was set up to discriminate between the two groups of genuine hazelnut oil and olive oil samples:

$$\text{rule} = \{[\text{scattering intensity (D1) at 1672 cm}^{-1}] - [\text{scattering intensity (D1) at 1658 cm}^{-1}]\}/[\text{scattering intensity (D1) at 1676 cm}^{-1}] \quad \text{(R1)}$$

D1 corresponds to the first derivative.

Figure 3B displays the values of the rule obtained from the FT-Raman spectra of the unsaponifiable matter of the training samples. All of the olive oil samples have a positive value ranging from 1.14 to 1.86 (mean = 1.57; SD = 0.14), although the range might have been slightly narrower (1.30−1.86) if the duplicates of two samples, which might be outliers, had not been taken into account. All of the hazelnut oil samples have negative values, ranging from −2.22 to −0.04 (mean = −0.62; SD = 0.45), with the exception of the duplicate of a Turkish refined hazelnut oil. These results indicate that the FT-Raman spectra of the unsaponifiable matter discriminate between OO and HAZ samples. The OO samples spiked with HAZ have, according to this decision rule, values ranging from 1.04 to 1.71 (mean = 1.46; SD = 0.20), so allowing the classification of some spiked samples outside the OO group.

The Fisher coefficient (*33*) was also used to detect MIR spectral zones that distinguish olive from hazelnut oils. The most important differences were found in the fingerprint region (1157−1543 cm^{-1}), which is characteristic of the stretching and bending vibrations of C−C and C−O groups of the molecules. In the case of the spectra of the unsaponifiable matter, a fraction composed of minor compounds (i.e., hydrocarbons, sterols, and terpenic and aliphatic alcohols among others), a decision rule (R2) was built with only three wavenumbers (i.e., 1438, 1461, and 1481 cm^{-1}):

$$\text{rule} = \{[\text{absorbance (D1 log 1/}R\text{) at 1438 cm}^{-1}] - [\text{absorbance (D1 log 1/}R\text{) at 1461 cm}^{-1}]\}/[\text{absorbance (D1 Log 1/}R\text{) at 1481 cm}^{-1}] \quad \text{(R2)}$$

D1 and R correspond to the first-derivative data and the reflectance, respectively.

The values of the rule allowed genuine hazelnut oils to be distinguished from genuine olive oils. All of the hazelnut samples had positive values (range = −0.01 to 0.12; mean =

0.03; SD = 0.21), whereas all of the olive oil samples had negative values (range = −1.01 to −0.24; mean = −0.6; SD = 0.21). These results show that the MIR spectra of the unsaponifiable matter discriminate between olive and hazelnut oil samples.

After this univariate study of the Raman and MIR spectra, the multivariate analyses were carried out by SLDA, which allowed the construction of discriminant equations based on the spectral information at discrete frequencies. For each spectral library constructed with the training samples, different SLDA discriminant models were built to discriminate between genuine hazelnut oils, genuine olive oils, and mixtures of them. **Table 1** presents the results of the SLDA multivariate analysis performed on FT-Raman and FT-MIR spectra of the entire oil and its unsaponifiable matter.

The SLDA model based on the Raman spectra of the entire oils selected 10 different Raman shifts. The Raman shifts included in the model were mainly characteristic of the vibration of trans and cis unsaturated groups (1693, 1674, 1670, 1659, and 3001 cm^{-1}) (*22*). The percentages of correct classification for the genuine olive oils, genuine hazelnut oils, and their mixture samples were 85.7, 100, and 86.7%, respectively. The SLDA model based on the FT-Raman spectra of the unsaponifiable matter selected Raman scattering intensities at seven different Raman shifts. The classification results show that 95.0, 100, and 97.5%, respectively, of the genuine olive oils, genuine hazelnut oils, and their mixtures were correctly classified. These Raman shifts belong to the 2950−3010 cm^{-1} region that is characteristic of the vibration of the unsaturated groups and the region (1674−1663 cm^{-1}), shown in **Figure 3A**, that has been attributed to the presence of squalene, a hydrocarbon that distinguishes the hazelnut oil from the olive oil (*8*).

The SLDA model based on the MIR spectra of the unsaponifiable matter selected eight wavenumbers (**Table 1**), most of them being from the fingerprint region (1500−900 cm^{-1}). Wavenumbers 980 and 987 cm^{-1} are related with the deformation vibration of the group C−H (trans −CH=CH− molecule), 1439 and 1446 cm^{-1} are related to the deformation vibration of the group C−H ($-CH_2$ molecule), 1628 and 1631 cm^{-1} are related to the stretching vibration of the group C=C (*cis*-CH=CH−), and 1728 cm^{-1} is highly correlated (0.96) with 1746 cm^{-1}, which is characteristic of the stretching vibration of C=O (−C=O molecule) (*22*, *34*). Only 4.5% of the genuine olive oils were incorrectly classified as adulterated (false positives), whereas the other samples were correctly assigned to their clusters. A similar model was designed with the spectra of the entire oil, but the results were worse (**Table 1**). This model has a high number of wavenumbers which might mean that it was low predictive and instable.

The next step was to validate the SLDA models with 44 blind samples plus other genuine olive and hazelnut oils. The described Raman and MIR models were checked with the spectra of the entire oil and its unsaponifiable matter. The best results were attained with the FT-MIR spectra of the unsaponifiable matter samples, although two false positives (a Turkish olive oil and a blend of various European olive oils) were detected. The limits of detection were not satisfactory for the blends with non-Turkish edible oils and blends of Turkish hazelnut oils and European olive oils (15%), whereas this limit was only 8% for blends obtained by mixing Turkish hazelnut and olive oils. **Figure 4** shows the results of the SLDA model based on the FT-MIR spectra of the unsaponifiable matter. The figure shows that the FT-MIR spectra of the unsaponifiable samples allow the discrimination between the pure olive oil samples and most of the adulterated samples.

There is a great interest in improving the knowledge of the spectroscopic bands of the minor compounds that are present in the olive oil unsaponifiable matter. Furthermore, more powerful discrimination mathematical procedures (i.e., ANN and Fuzzy Logic) are being implemented as well.

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