Comparison of Near-Infrared, Fourier Transform-Infrared, and Fourier Transform-Raman Methods for Determining Olive Pomace Oil Adulteration in Extra Virgin Olive Oil

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ABSTRACT: The adulteration of extra virgin olive oil with cheaper oils is a major problem in the olive oil market. In this study, near-infrared, mid-infrared, and Raman spectroscopic techniques were used to quantify the amount of olive pomace oil adulteration in extra virgin olive oil. The concentration of olive pomace oil in extra virgin olive oil was in the range between 0 and 100% in 5% increments by weight. Of the methods studied, Fourier transform-Raman spectroscopy gave the highest correlation with a correlation coefficient of 0.997 and a standard error of prediction of 1.72%. The spectroscopic techniques have the potential to become a tool for rapid determination of adulteration in extra virgin olive oil, because they are easy to use and cost-effective.

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KEY WORDS: Adulteration, chemometrics, mid-infrared, near-infrared, olive oil, Raman spectroscopy.

Olive oil not only contains a fine aroma and a pleasant taste but also is known for its health benefits (1). Compared to other seed oils, olive oil has low levels of saturated (~16%) and high levels of monounsaturated (~70%) fatty acids. The quality of olive oil ranges from the high-quality extra virgin olive oil to the low-quality olive pomace oil (2). Major markets for olive oil are in Europe, the United States, and Canada. Extra virgin olive oil is mechanically extracted from the olive (*Olea europaea sativa* H.) fruit without any thermal or chemical treatments. Olive pomace oil, a fully refined olive oil, is extracted by solvent extraction processes from olive pomace. The high-quality/-grade olive oil (e.g., extra virgin olive oil) fetches a higher price because of its taste and aroma compared to the lower quality olive pomace oil. Hence, there is a great temptation to use lower grades of olive oil, such as olive pomace oil, as an adulterant in extra virgin olive oil to extend and increase profits. The authentication of extra virgin olive oil and its adulteration with lower-grade oils are very serious problems in the olive oil industry and deserve attention.

Free fatty acid (FFA) expressed as oleic acid, one of the critical components in olive oil, can be used to assess the quality of olive oil. The International Olive Oil Council states that FFA in extra virgin olive oil should be less than 1%, while the FFA content in lower-grade olive oil could be as high as 3% (2). However, lower-grade olive oil could be processed to reduce the FFA content, and the resulting product could be mislabeled as "extra virgin olive oil." A rapid method to detect such practices in the olive oil business is important for quality control and labeling purposes. Chromatography (3) and ^{1}H and ^{13}C nuclear magnetic resonance spectroscopy (4,5) have been used to study olive oil adulteration. The methods have the potential to clearly trace the minor components of the adulterants (e.g., fatty acids, trilinolein, equivalent carbon number-43 sterols, and hydrocarbons) in extra virgin olive oil. However, these techniques are expensive, complex, laborious, time-consuming, and require skilled personnel and sophisticated instrument capability. There is a need to develop a simple, cheap, and rapid alternative to determine adulterants in extra virgin olive oil.

Vibrational spectroscopy combined with chemometric methods is an emerging analytical technique for adulterant determination in olive oil. Vibrational spectroscopy includes infrared (IR) and Raman techniques. They are powerful and rapid analytical techniques that provide not only quantitative information but also qualitative information (e.g., chemical structure of functional groups) of the samples. The IR spectrum is obtained by a change in the molecular dipole moment during vibration, while the Raman spectrum is obtained by a change in polarizability during the vibration (6). For example, the C=O (CO_2) and O–H (H_2O) stretch, polar functional groups, have strong absorption in the IR spectrum, while the C=C stretch, nonpolar functional group, has strong Raman scattering in the Raman spectrum. The C–H stretch has a strong absorbance in the IR spectrum and also a strong Raman scattering band in the Raman spectra, while O–H stretch has a strong absorbance in the IR spectrum but a very weak absorbance in the Raman spectrum. The peaks/bands in the IR and Raman spectra at a specific frequency/wavenumber are characteristic of chemical groups that constitute the components in the samples.

The peaks in the near-infrared (NIR) region (1100 to 2500 nm or 9091–4000 cm^{-1}) are broad and weak due to combinations and overtones of functional groups of sample chemical constituents, and hence NIR is mostly used in quantitative analysis. The advantages of NIR spectroscopy are its rapid and nondestructive measurement capability and on-line application potential for direct process control. NIR has been used in many areas of food and pharmaceutical industries for quality evaluation. NIR spectroscopy was first applied to determine the presence of adulterants (corn oil, sunflower oil,

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and raw olive residue oil) in olive oils (7). Principal component analysis (PCA) was used to build the calibration model to predict the adulterant contents. Results demonstrated that NIR is a possible tool to determine adulteration in olive oil, but the prediction of the adulterant type was not very accurate. A further study using NIR spectroscopy identified adulterants (sunflower, rapeseed, and soybean oils) by PCA and their levels in olive oils by partial least squares (PLS) (8). A discriminant analysis equation was developed to correctly identify the type of seed oils in extra virgin olive oil with 90% accuracy. Another related study (9) includes screening and authentication of extra virgin olive oils based on their geographical origins using artificial neural networks (ANN). Other applications include the classification of vegetable oils, such as soybean, corn, cottonseed, olive, rice bran, peanut, rapeseed, sesame, and coconut oils (10,11).

Unlike NIR, most of the peaks in the mid-infrared (MIR) region (4000–400 cm^{-1}) are narrow and sharp and are due to fundamental vibrations of the molecules. Compared to dispersive NIR, Fourier transform-infrared (FTIR) spectroscopy in the MIR region has a higher signal/noise ratio and resolution. FTIR has been used to study edible oils and oxidation of fat (12). The potential of FTIR with attenuated total reflectance (ATR) accessory to estimate the level of adulteration in olive oil has been examined by Lai *et al*. (13,14). PCA, PLS, and discriminant analysis were applied to interpret spectral data. From discriminant analysis, 57 out of 61 samples were correctly classified into the 13 oil samples considered, based on five principal component scores (14). Other studies included the determination of refined olive oil and walnut oil (13), cheaper seed oils (15), safflower and sunflower oils (16), and vegetable oil (17) concentration as adulterants in extra virgin olive oil.

Fourier transform (FT)-Raman spectroscopy can be regarded as a complementary method to the IR technique for analysis (18). Like FTIR spectroscopy, FT-Raman can also provide information on the functional/chemical groups for qualitative and quantitative characterization. By using linear discriminant analysis and ANN, a comparison study of FTIR and FT-Raman techniques for the authentication of olive oil gave a classification accuracy of 100% using FTIR spectroscopy and 93.1% using FT-Raman spectroscopy (19). The adulteration detection limits were 5% for the adulterants of seed oils and refined olive oil for IR data, while they were 45% for refined olive oil and 5% for seed oils for Raman data. In an FT-Raman study on detecting adulteration, lower detection limits with R^2 value of 0.964 were obtained using PCA (20). Adulterants considered were olive pomace, soybean, and corn oils at levels of 0, 1, 5, and 10%. FT-Raman spectroscopy was also used to classify olive oil from other vegetable oils and animal fats (21,22). It should be noted that only limited concentrations were used. The Raman intensities at 3013, 1663, and 1264 cm^{-1} had a high correlation with unsaturated, monounsaturated, and polyunsaturated fatty acids, respectively, in the oils.

A detailed spectroscopic study of the varying levels of adulteration and a comparative evaluation of the different techniques have not been attempted. In this study, a lower grade of olive oil (pomace olive oil) was added to extra virgin olive oil at 5% concentration increment levels, and IR (NIR and MIR) and Raman techniques were applied to quantify the extent of the individual components in the binary mixtures. Furthermore, the first application of FTIR-photoacoustic spectroscopy (PAS) for the detection of adulteration in olive oil is presented and compared with other IR and Raman methods. Only one study (23) on the use of FTIR-PAS for oil analysis has been reported in the literature. The two objectives of this research are (i) to compare the spectroscopic determinations of olive pomace adulteration using of NIR, FTIR, and Raman methods and (ii) to demonstrate the potential of a new FTIR-PAS method for oil characterization.

MATERIALS AND METHODS

Materials. Olive pomace oil was obtained from the North American Olive Oil Association, and extra virgin olive oil (Bertolli USA, Secaucus, NJ) was purchased from Sam's Club (State College, PA). The percentage of pomace olive oil in extra virgin olive oil is generally less than 15%, but for calibration purposes, olive pomace oil was added to extra virgin olive oil in the concentration range between 0 and 100% in 5% increments for spectroscopic measurement. A total of 21 different concentrations was considered, and each experiment was replicated four times.

NIR measurement. The DA 7000 Visible/NIR dispersive spectrometer (Perten Instruments Inc., Springfield, IL) with silicon and InGaAs diode arrays was used to determine olive pomace oil in extra virgin olive oil. A fiber-optic probe was attached to the spectrometer for data collection as described by Oskin (24). A white PVC casing 2 in. in diameter was used to keep a 2-in. distance between the sample and the probe. The oil sample was placed in a Petri dish and the spectra were collected. For each spectrum, an average of 30 scans was recorded over the 400–1700 nm wavelength range at 10-nm intervals. A white Spectralon plate (Labsphere, Inc., North Sutton, NH) was used as a background. Each test was set up automatically to obtain three measurements.

FTIR-ATR measurement. The Nexus 870 FTIR spectrometer (Nicolet Instrument Corp., Madison, WI) with a deuterated tri-glycine sulfate detector was used for experiments. A ZnSe ATR sampling accessory from Spectra-Tech (Shelton, CT) was used for ATR measurements. ATR spectra were collected at 256 scans/sample at 4 cm^{-1} resolution. Before scanning each sample, the background spectrum was taken with an empty ATR crystal and stored in the computer. The sample was then poured on the ATR ZnSe crystal for measurement. After each measurement, the ATR crystal was thoroughly cleaned with 1% Triton X-100 solution (Aldrich Chemical Co., Milwaukee, WI), followed by a hexane (Aldrich Chemical Co.) wash. The washed crystal was then rinsed with distilled water, wiped with cotton, and dried using nitrogen gas after each measurement.

FTIR-PAS measurement. FTS 6000 research-grade FTIR spectrometer (Bio-Rad Laboratories, Cambridge, MA) with a

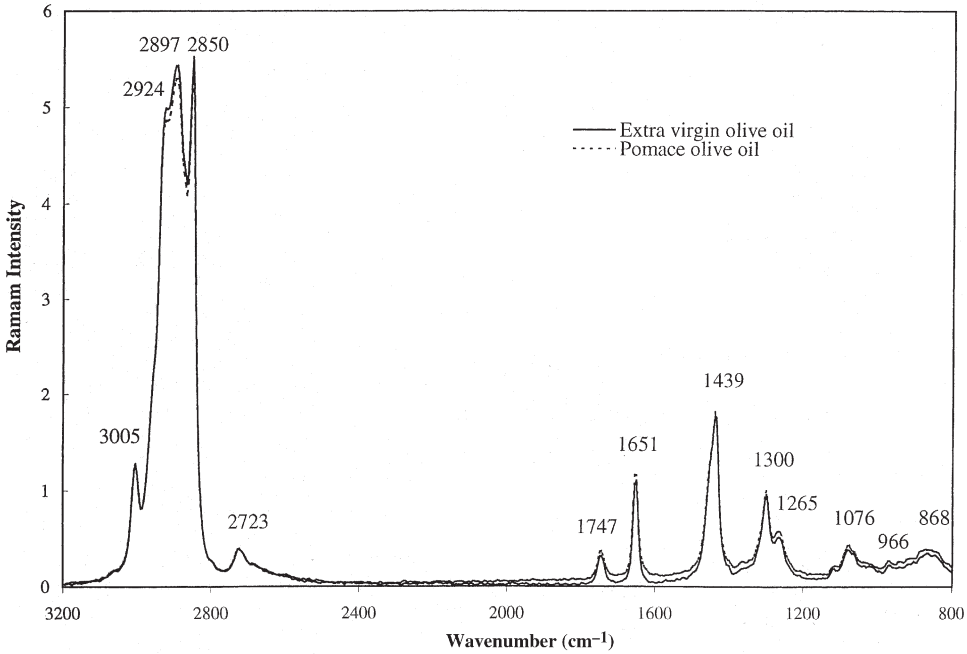

FIG. 1. Plot of Fourier transform (FT)-Raman spectra of extra virgin olive oil and olive pomace oil.

helium-purged photoacoustic (PA) detector (MTEC Photoacoustics Inc., Ames, IA) was used for collecting the PA spectra. Carbon black was used as the background. Half of the stainless-steel cup (10 mm in diameter and 3 mm in depth) was filled with oil sample and placed in the PA detector for analysis. The PA detector was purged with helium for 5 min and then sealed. Spectra of samples were obtained at a resolution of 4 cm^{-1} and scan number of 256/sample.

FT-Raman measurement. The Nexus FT-Raman spectrometer (Nicolet Instrument Corp.) with 1064 nm excitation, an InGaAs detector, and 0.5 W power was used in the experiment. The oil samples analyzed were poured into a glass test tube (6 mm in diameter) and placed in a sample holder for FT-Raman measurement. The spectra were collected using 256 scans/sample at 8 cm^{-1} resolution.

Data analysis. Spectral data from the individual instrument measurements were divided into a calibration set (75%) and validation set (25%). PLS regression was applied to determine the concentration of olive pomace oil in extra virgin olive oil. PLSplus/IQ from GRAMS/32 (Galactic Industries Co., Salem, NY) was used for PLS model building and statistical analysis. For PLS model construction, the first step was to select the spectral region that best represents the analyte to be determined. Multiplicative scatter correction (MSC) was selected to correct for the variation caused by light-scattering. The model was chosen based on the optimal number of factors, the minimum value of the predicted residual sum of squares (PRESS), and the maximum value of the correlation coefficient (R^2). The resulting models from different methods were then evaluated based on the number of factors, standard error of prediction (SEP), standard error of cross-validation (SECV), and R^2.

RESULTS AND DISCUSSION

FT-Raman. Figure 1 compares the spectra of extra virgin olive oil and olive pomace oil. The assignment of major peaks in the FT-Raman spectra is listed in Table 1 using previous studies (21,22,25,26), as the basis. Extra virgin olive oil has a lower intensity between 3000 and 2800 cm^{-1}, but olive pomace oil has a higher intensity beyond 2000 cm^{-1}. Normally, *trans* fatty acids (C=C), labeled as saturated fat content by the United States Food and Drug Administration (FDA), can be found in most of the seed oils and animal fats but not in extra virgin olive oil (20; ≤0.1%). Olive pomace oil may contain the *trans* fatty acids (2; ≤0.75%) and hence show Raman scattering bands corresponding to this functional group. *Trans*

TABLE 1
Chemical Assignment of Bands in the FT Raman Olive Oil Spectra[a]

Wavenumber (cm^{-1})	Molecule	Vibration of mode	Intensity
3005	*cis* RHC=CHR	=C–H stretching (sym)	m
2924	$–CH_2$	C–H stretching (asym)	sh
2897	$–CH_3$	C–H stretching (sym)	s
2850	$–CH_2$	C–H stretching (sym)	s
2723	$–(CH_2)_n–$	C–H stretching	w
1747	RC=OOR	C=O stretching	w
1651	*cis* RHC=CHR	C=C stretching	m
1439	$–CH_2$	C–H bending (scissoring)	s
1300	$–CH_2$	C–H bending (twisting)	m
1265	*cis* RHC=CHR	=C–H bending	sh
1076	$–(CH_2)_n–$	C–C stretching	vw
968	*trans* RHC=CHR	C=C bending	w
868	$–(CH_2)_n–$	C–C stretching	w

[a]FT: Fourier transform, s: strong, m: medium, w: weak, sh: shoulder, vw: very weak, sym: symmetry, asym: asymmetry.

TABLE 2
Results of Calibration and Validation for NIR, MIR, FT Raman Techniques[a]

	Calibration				Validation	
	Factor numbers	PRESS	R^2	SECV	R^2	SEP
FT Raman	1	0.031	0.995	2.23%	0.997	1.72%
FTIR-ATR	11	0.122	0.981	4.74%	0.991	3.28%
FTIR-PAS	4	0.135	0.982	4.45%	0.990	6.51%
NIR	13	0.408	0.990	3.48%	0.990	3.27%

[a]FTIR-ATR, Fourier transform infrared-attenuated total reflectance; PAS, photoacoustic spectroscopy; NIR, near infrared; PRESS, predicted residual sum of squares; R^2, correlation coefficient; SECV, standard error of cross-validation; SEP, standard error of prediction; MIR, mid-infrared; see Table 1 for other abbreviation.

C=C bonds can be presented as a stretch at ~1670 cm^{-1} and bend at ~966 cm^{-1} in the Raman spectrum. The stretch of *trans* C=C could not be observed (Fig. 1) probably due to the overlap of a much larger and broader band nearby at 1651 cm^{-1} (*cis* C=C stretch), caused by the high concentration of unsaturated fatty acids in olive oil. However, a very weak band attributable to bend was observed at 968 cm^{-1} (Fig. 1). The intensity of the *trans* C=C bend-related chemical groups is higher in olive pomace oil than extra virgin olive oil.

When the spectra around 968 cm^{-1} (1000 to 950 cm^{-1}) were used for quantitative analysis, an R^2 value of about 0.5 was obtained. In this study, the entire spectral region from 4000 to 400 cm^{-1} was selected for analysis. Table 2 summarizes the prediction results using the different spectroscopic methods. An R^2 value of 0.995 (SECV = 2.23%) for the PLS calibration model and R^2 value of 0.997 (SEP = 1.72%) for the validation model were obtained, implying that the model is reliable (Fig. 2).

FTIR-ATR. The spectra of different concentrations of olive pomace oil in extra virgin olive are shown in Figure 3 with the assignments of major peaks (23) (Table 3). The IR and Raman spectra have their similarities and dissimilarities. For example, the C–H stretch has very strong bands or intensity in the region between 3000 and 2800 cm^{-1} in both IR and Raman spectra, while the C=O stretch has a very intense absorbance at 1745 cm^{-1} in IR spectra (Fig. 4) but a weak Raman band (Fig. 1). Similar to FT-Raman spectra, the *trans* C=C component in oil samples was observed at 968 cm^{-1} while the peak was very weak. The stretching of *cis* C=C at

TABLE 3
Chemical Assignment of Bands in the FTIR Olive Oil Spectra (23)[a]

Wavenumber (cm^{-1})				
ATR	PAS	Molecule	Vibration of mode	Intensity
3008	3005	*cis* RHC=CHR	=C–H stretching (sym)	m
2954	2955	$-CH_3$	C–H stretching (asym)	sh
2926	2928	$-CH_2$	C–H stretching (asym)	s
2854	2857	$-CH_2$	C–H stretching (sym)	s
1745	1749	RC=OOR	C=O stretching	s
1653	1653	*cis* RHC=CHR	C=C stretching	w
1466	1466	$-CH_3$, $-CH_2$	C–H bending (scissoring)	s
1419	1420	*cis* RHC=CHR	=C–H bending (rocking)	w
1379	1377	$-CH_3$	C–H bending	m
1240	1240	$-CH_2$	C–H bending	w
1163	1165	$-CH_2$	C–H bending	s
		–CO–O–	C–O stretching	
1120	1119	–CO–O–	C–O stretching	w
1099	1099	–CO–O–	C–O stretching	w
1034	1034	–CO–O–	C–O stretching	w
968	968	*trans* RHC=CHR	C=C bending	vw
723	723	$-(CH_2)_n-$	C–H bending (rocking)	s

[a]See Tables 1 and 2 for abbreviations.

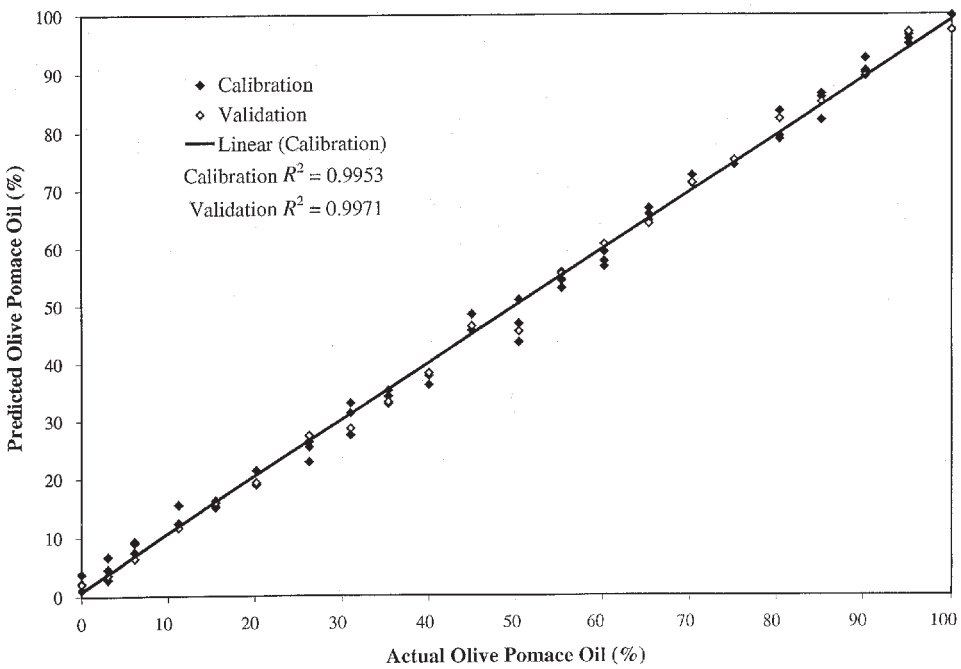

FIG. 2. FT-Raman predicted and actual concentration plot for calibration and validation models. R^2, correlation coefficient; see Figure 1 for other abbreviation.

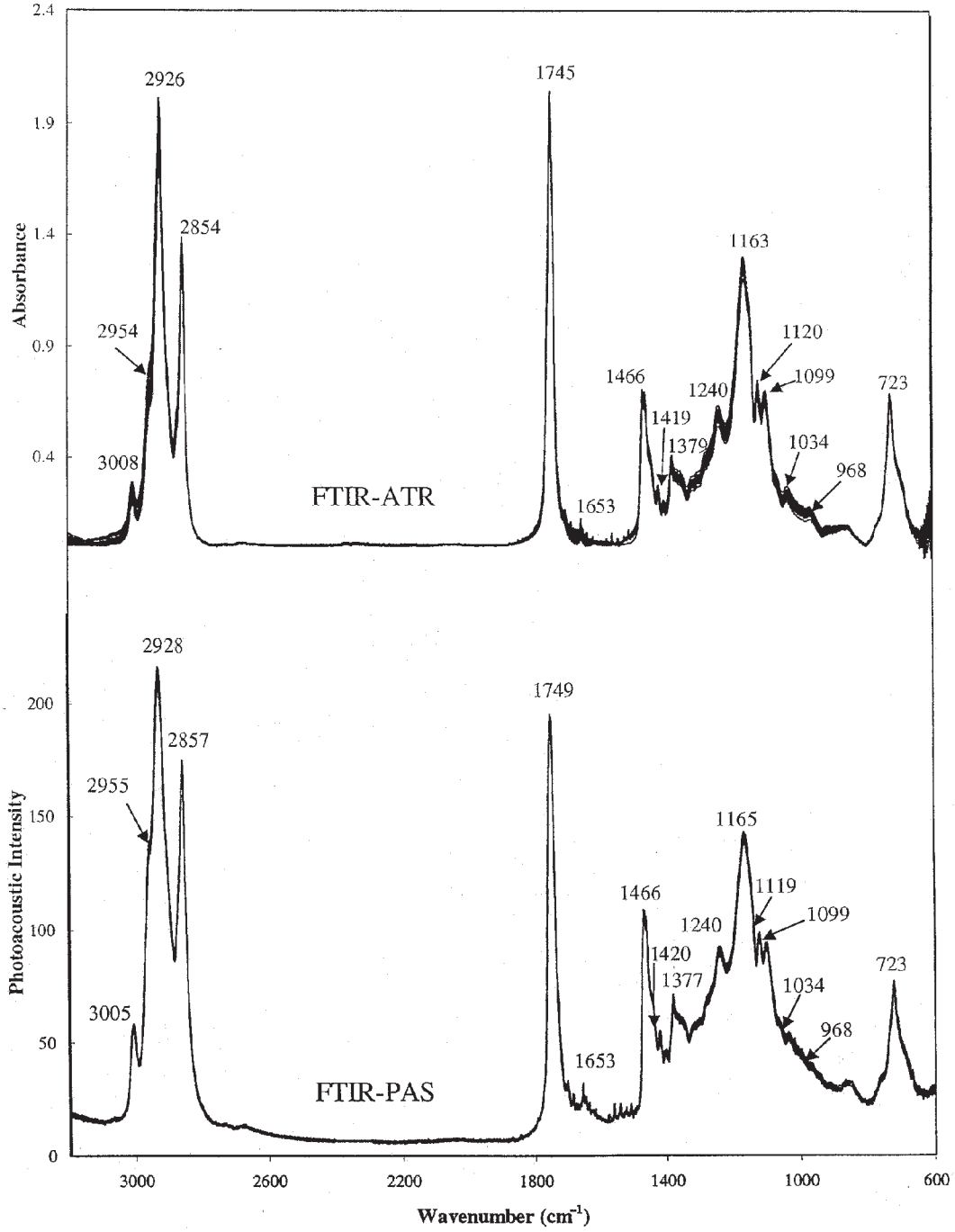

FIG. 3. Plot of Fourier transform infrared-attenuated total reflectance (FTIR-ATR) and FTIR-photoacoustic spectroscopy (PAS) spectra of different concentrations of olive pomace oil in extra virgin olive oil.

1653 cm^{-1} in the IR spectra (Fig. 3) was weak but showed a strong Raman band (Fig. 1). This is due to the complementary nature of IR and Raman methods.

There is a significant difference in the concentration of FFA between extra virgin olive oil and olive pomace oil. The entire IR region from 4000 to 600 cm^{-1} was selected for building the PLS calibration model. A minimal PRESS value of 0.122 was obtained with 11 factors in the model. R^2 values of 0.981 (SECV = 4.74%) and 0.991 (SEP = 3.28%), respectively, were obtained for calibration and validation data (Table 2). The results of this study were different from the results of Marigheto *et al.* (19), which reported that FTIR spectroscopy was better suited for classification and quantification of oils than Raman.

FTIR-PAS. FTIR-PAS is a nondestructive analysis method and does not require any sample preparation. FTIR-PAS spectrum (Fig. 3) of olive pomace oil in extra virgin olive oil was very similar to FTIR-ATR. The wavenumbers of major peaks in FTIR-PAS spectra were very close to those in FTIR-ATR spectra (Table 3). The peak at 968 cm^{-1} attributable to *trans*

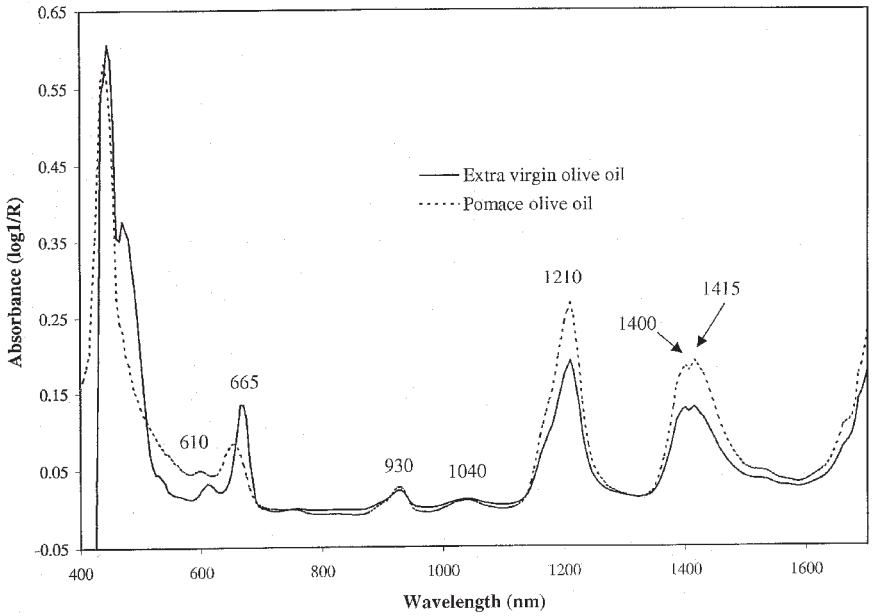

FIG. 4. Plot of near infrared spectra of extra virgin olive oil and olive pomace oil.

C=C bend was also captured in the PAS spectra but was weaker than that in the ATR spectra. Generally, FTIR-PAS can serve as an alternative to FTIR-ATR. The FTIR-PAS sample cell can be easily cleaned after analysis compared to the ATR crystal, which requires an elaborate cleaning procedure to avoid the "memory effect" of oil sample on the surface of the ATR crystal (12).

To build the PLS calibration model based on FTIR-PAS spectra, the regions 3042 to 2823 cm^{-1} and 1778 to 634 cm^{-1} were selected, because the chemical/functional groups of FFA are contained in this region (27). The best calibration model was determined corresponding to a minimum PRESS value of 0.135 and a factor number of 4 (Table 2). The respective R^2 values of 0.982 (SECV = 4.45%) and 0.990 (SEP = 6.51%) were obtained for calibration and validation.

NIR. NIR spectra of extra virgin olive oil and olive pomace oil are shown in Figure 4. The spectral region covers both the NIR and visible regions, with the absorbance at 610 nm denoting an orange color and the absorbance at 665 denoting a red color. The peaks of NIR attributable to the combinations and overtones are shown in Table 4 with the band assignment. Olive pomace oil has a higher absorbance intensity in the region between 1100 and 1700 nm than extra virgin olive oil (Fig. 4). For building the PLS calibration model, specific FFA regions (470 to 690, 1145 to 1265, and 1355 to 1500 nm) (28) of the NIR spectra were selected because of their high correlation with the concentrations of olive pomace oil. The model with the minimal PRESS value of 0.4080 corresponding to 13 PLS factors was chosen for calibration. R^2 values of 0.990 (SECV = 3.48%) and 0.990 (SEP = 3.27%) were obtained for the calibration and validation data (Table 2).

Comparison of the prediction models from the different spectroscopic methods indicated that FT-Raman spectroscopy gave the highest correlation (R^2 = 0.997) with the lowest prediction error (SEP = 1.72%). However, NIR and MIR techniques also provided good predictions with an R^2 value greater than 0.99. The nondestructive evaluation, ease of operation, and fast determination merit the use of IR and Raman techniques for adulterant determination studies. The techniques presented could also be used to study the adulteration of extra virgin olive oil with other lower-quality oils.

TABLE 4
Chemical Assignment of Bands in the NIR Olive Oil Spectra[a]

Wavelength (nm)	Molecule	Vibration of mode	Intensity
930	$-CH_2$	C–H stretching 3rd overtone	w
1040	$-(CH_2)_n-$	C–H stretching and bending	w
1210	$-CH_2$	C–H stretching 2nd overtone	s
1400	$-(CH_2)_n-$	C–H stretching and bending	s
1415	$-CH_2$	C–H stretching 1st overtone	s

[a]See Tables 1 and 2 for abbreviations.

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