

## Characterization and rapid detection of adulterations in sesame oil using FT-MIR and PCA-LDA

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### Abstract

Sesame oil has an important place in human nutrition, medicinal, pharmaceutical, industrial and agricultural uses. The objective of this research work was to use Attenuated total reflectance-Fourier transform mid-infrared (ATR-FTMIR) spectroscopy coupled with chemometrics for the detection of the adulteration of sesame oil. Adulteration with sunflower oil, soybean oil or colza oil is one of the most difficult to detect due to the similar composition of them and sesame oil. Adulterations of sesame oil with different percentages of sunflower oil, soybean oil and colza oil were measured using ATR-FTMIR spectroscopy. The spectral data were subjected to a preliminary derivative elaboration based on the Savitzky–Golay algorithm to reduce the noise and extract a largest number of analytical information from spectra. Linear discriminant analysis (LDA) was adopted as classification method, and Principle component analysis (PCA) was employed to compress the original data set into a reduced new set of variables before LDA. The detection results indicated that the discriminant model built by PCA-LDA method could identify sesame oil adulterations in the 0–40% weight ratio range of edible oils, with an accuracy value of 94.64%. This work shows that PCA and LDA are useful chemometric tools for the multivariate characterization and discrimination of sesame oil adulteration with seed oils

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## 1. Introduction

The identification and authentication of products are current topics in food and feed science for both sides: on the one hand consumer protection authorities and on the other hand producers and dealers. Since the beginning of food trade, incidents concerning adulterations of relevant products are well known [1]. In fact, adulteration is a major issue in the high quality oils market. The adulteration of food products presents a huge importance for consumers, food processors, regulatory agencies and industries, done for economic reasons. It involves replacing or dilution of high cost ingredients with lower and cheaper ones [2, 3]. Actually, sesame oils can be considered expensive compared to other vegetable oils, which makes its marketing a bit difficult in some countries. The authentication of sesame oil (SO) has become very important due to the possible adulteration of SO with cheaper plant oils such as colza (CO), soybean (SBO) and sunflower (SFO) oils. The detection of adulteration is more difficult, especially when the adulterant has similar chemical composition to that of the original oil [4]. In addition, one trend of analytical chemistry is the development of new vanguard systems to address the high demand of quality information society requires [5]. Today, the application of Fourier transform infrared (FTIR) spectroscopy has increased in food studies, and particularly has become a powerful analytical tool in the study of edible oils and fats [6]. For qualitative analysis, the FT-MIR spectroscopy has more applications, because the 'fingerprints' of functional groups can be displayed narrowly and intensely in the MIR region ( $4000\text{--}400\text{ cm}^{-1}$ ). Fourier transform infrared (FTIR) spectroscopy with attenuated total reflectance (ATR) or transmission cell accessories has been used in several studies concerning to authentication, characterization, identification or classification of fats and oils [7, 8, 9, 10, 11, 12, 13]. FTIR data have been often treated with multivariate analytical techniques to develop methods of classification and characterization, through the building of relative models. This approach has demonstrated to be very useful in many applications, due to the ability in achieving the spectral resolution of the FTIR signals [14], and its simplicity, rapidity, and ease of sample preparation [15]. To the authors' knowledge, nowadays, there are not official methods to verify the quality and authenticity of sesame oil. Then, there is a need to develop analytical methods to detect and quantify the amount of adulterations in sesame oil. The aim of this paper was to characterize and classify pure sesame oils and adulterated ones by using a fast and suitable analytical technique. In this case, we tried to develop a new application of the ATR-FTIR coupled to multivariate statistical analysis methods, such as PCA and typical classification techniques, LDA, because it has been successfully used in the quality control of food [16, 17, 18]. In fact, this approach is a rapid, inexpensive and non destructive authenticity measuring tool, useful to identify and detect sesame oil quality (pure or adulterated). Methods involving Fourier transform mid infrared (FT-MIR) spectroscopy combined with chemometric tools were developed for classification of four classes: pure SO and binary mixtures SO-SFO, SO-SBO or SO-CO in the adulteration range of 0–40 % weight ratios.

## 2. Experimental

### 2.1. Sampling

Forty authentic samples of sesame oil (SO) extracted by soxhlet with hexane as solvent was personally taken from different sources in Morocco. Colza oil, soybean oil, sunflower oil were used as adulterants and were purchased in a local supermarket. For the adulteration study, oil samples were prepared by mixing sesame oil (SO) with colza oil (CO), soy bean oil (SBO), and sunflower (SFO) in the range of 0–40%. The samples were stored in the dark at ambient temperature until analysis. In fact, we created four classes of samples: pure SO and binary mixtures SO-SFO, SO-SBO or SO-CO. Each class includes 40 samples. There were 160 samples in total, among which 104 samples were randomly for establishing principal component analysis and linear discriminant analysis models. Other 56 samples were used for testing the models.

## **2.2. Instrumentation and analysis**

ATR-FTMIR spectra were obtained using a Vector 22 Bruker FTIR Spectrophotometer equipped with an attenuated total reflectance accessory (ATR single-reflexion, Diamond, incident angle 45°, Pike Miracle, Pike Technologies, Madison, USA) with DTGS detector, Globar (MIR) Source and KBr Germanium separator. The measurements were directly carried out by putting oil samples on ATR surface, provided with a diamond crystal, at controlled room temperature (25°C) in MIR region of 3000–600 cm<sup>-1</sup>, by accumulating 90 scans with the resolution of 4 cm<sup>-1</sup>. The data were handled with OPUS logiciel. The software (Opus 4.0 MSD) fitted to the infrared spectrometer Fourier transform used in this study allows the automatic acquisition of the spectra without any form of computer manipulation may impair the quality of results. The Fourier transform is automatically calculated by the software prior to the acquisition of spectra. Air was taken as reference for the background spectrum before each sample. Between spectra, the ATR plate was cleaned in situ by scrubbing with ethanol solution, enabling to dry the ATR.

## **2.3. Chemometrics**

The chemometric evaluation was performed by the following multivariate techniques: principal component analysis (PCA) and linear discriminant analysis (LDA). Both multivariate analyses show effective results in various applications [16, 19, 20, 21]. In this work, we used PCA and LDA as methods for detecting sesame oil adulterations according to ATR-FTMIR analysis.

The PCA and LDA were performed using The Unscrambler X version 10.2 (CAMO, Oslo, Norway).

### **2.3.1. Principal Component Analysis (PCA)**

Principal component analysis (PCA) is a standard tool in chemometric techniques for data compression. Then, it simplifies the working with multivariate data sets like MIR and NIR data sets and features extraction. PCA is a method of data reduction that constructs new uncorrelated variables called principal components (PCs) that are linear combination of the original measurement variables [22, 23, 24]. So, PCA is a linear projection method, based on variance, reduces the dimensionality of the original data matrix retaining the maximum amount of variability [25]. Its major objective is to find out trends in the variables and samples [26]. Results of PCA are visualised by scores and loading plots. The scores vectors describe the relationship between the samples and allow checking if they are similar or dissimilar, typical or outlier, while the loadings vectors describe the importance of each variable [27, 28].

In this study, PCA was used to reduce the dimensionality of spectral data of sesame oils samples while retaining as much information as necessary. This thanks to the linear combinations of the original independent variables that are used to explain the maximum variance of data set.

### **2.3.2. Linear Discriminant Analysis (LDA)**

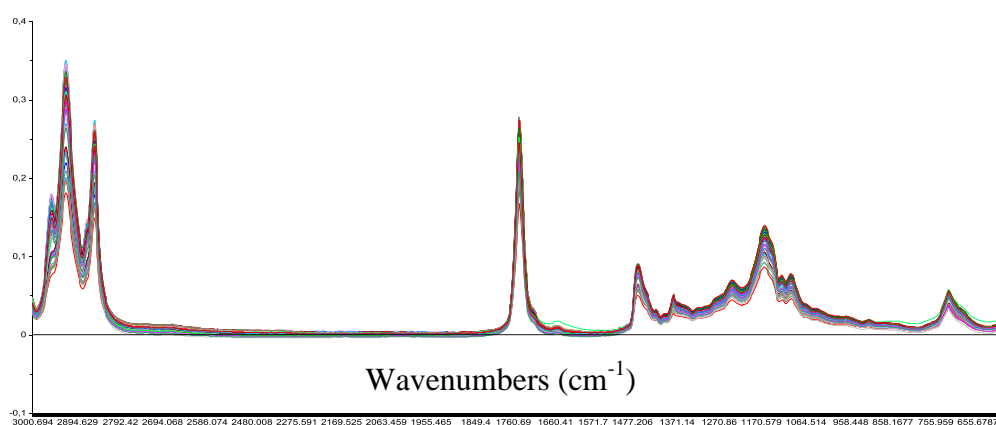
Linear Discriminant Analysis (LDA) is a supervised classification method to discriminate groups of samples as a function of one or several combinations of experimental Variables [29, 30]. Each combination is called a “Discriminant Function” (DF), which resembles the idea of the PCA [31]. LDA is a projection technique to find directions of maximum separation from a set of samples for which class membership is known in order to be able to predict the class membership of unknown samples [5, 32, 33]. Description of the LDA algorithm can be found in Ref. [34] in detail. In the other side, the FTIR spectra have highly multivariate data and number of samples is moderate, and thereafter, the LDA classification model may become unstable. This problem can be overcome and a reduction in data dimension is possible by decomposing and projecting the samples onto a smaller number of orthogonal components found by PCA. Then, Combination of PCA and LDA has been proposed for classification and

discrimination of the studied samples [35]. Application of PCA before LDA is reported in the literature [36]. The performance of the calibration model was estimated from the percentage of correctly classified samples (%CC) [37].

### 3. Results and discussion

#### 3.1. Spectral diversity

Figure 1 shows MIR spectra of sesame oil samples at frequency region of 3000–600  $\text{cm}^{-1}$ . The region between 4000 and 3000  $\text{cm}^{-1}$  is removed due to the water which is instrumental noise and brings useless information [38]. The assignment of functional groups responsible for IR absorption, according to literature, is as follows:  $\sim 3000 \text{ cm}^{-1}$  (trans  $=\text{C}-\text{H}$  stretch), 2920 and 2852  $\text{cm}^{-1}$  (symmetrical and asymmetrical stretching of  $-\text{CH}_2$ ), 1743  $\text{cm}^{-1}$  ( $-\text{C}=\text{O}$  stretch), 1463  $\text{cm}^{-1}$  ( $-\text{CH}_2$  bending), 1377  $\text{cm}^{-1}$  ( $-\text{CH}_3$  bending), 1237  $\text{cm}^{-1}$  ( $-\text{C}-\text{O}$  stretch), 1161  $\text{cm}^{-1}$  ( $-\text{C}-\text{O}$  stretch;  $-\text{CH}_2$  bending), 1118  $\text{cm}^{-1}$  ( $-\text{C}-\text{O}$  stretch), 1096  $\text{cm}^{-1}$  ( $-\text{C}-\text{O}$  stretch), and 722  $\text{cm}^{-1}$  (cis- $\text{CH}=\text{CH}-$  bending out of plane) [39,40, 41,42]. bands of holocellulosic materials in the 1200 - 900  $\text{cm}^{-1}$  region [43].



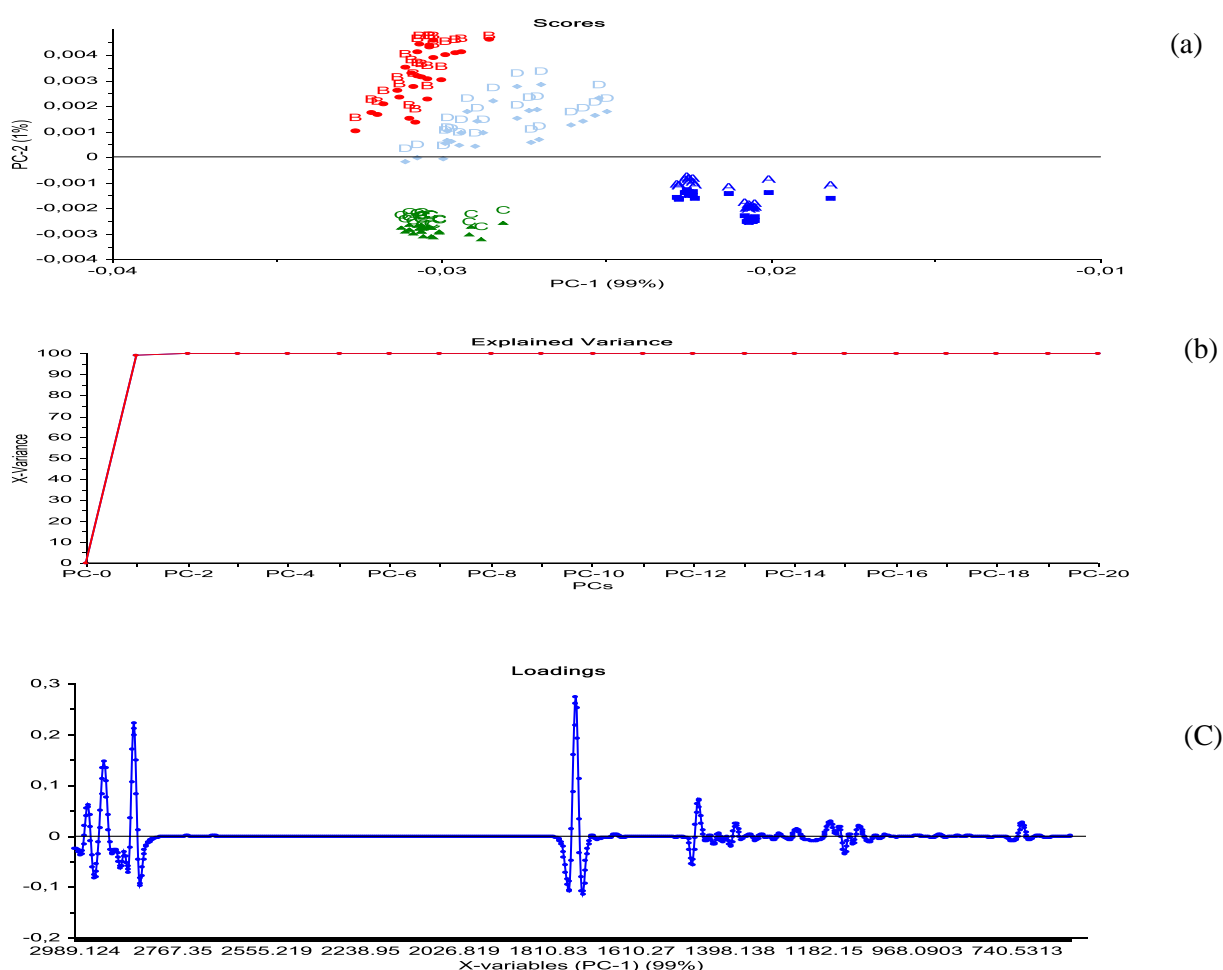
**Figure 1:** ATR-FTMIR spectra of the three binary mixtures (SO-SFO, SO-SBO and SO-CO) and pur sesame oil samples in the spectral range 3000–600  $\text{cm}^{-1}$

Fourier transform infrared (FT-IR) spectra obtained for 104 samples of sesame oil are simulated by visual inspection. This is due to the similar chemical composition of SO and adulterants oils (SFO, SBO and CO) [4, 44]. Multivariate calibration methods appeared then to be ideal to provide an effective solution, as they allow extracting of unspecific analytical information from the full-spectra or large regions of them. In addition, a preliminary treatment of the data seemed necessary to extract better analytical information. At the same time, a data pre-treatment was considered useful to minimize instrumental problems as baseline fluctuation or noise. The spectral data were first derived, with the algorithm developed by Savitzky and Golay [45] to remove unwanted spectral variations as offsets. The best improvement in data variance was reached when the derivative function through the Savitzky Golay algorithm was used. Best results were obtained by fixing the following parameters: 2nd order, number of smoothing points 13 and polynomial order 2.

#### 3.2. PCA modeling

A non-supervised multivariate method, the principal component analysis was used to analyse the spectroscopic data of sesame oil samples (pure and adulterated) and to evaluate the capability of discrimination of the samples according to

their quality. PCA allows determining the main features of the spectra, to compare them and to highlight links between the descriptive variables (the absorbance at different wavelengths) [46]. The PCA model was built with the NIPALS algorithm, by considering all frequency intervals 3000–600  $\text{cm}^{-1}$ , as X variables, and the model was validated by full Cross validation. With full Cross validation, the same samples are used both for model estimation and testing: leaves out only one sample at a time [47]. Results of PCA are visualized by scores and loading plots. Scores plot (Figure 2.a) was constructed to observe principal groupings among observations. Loading plot (Figure 2.c) indicates the importance of each variable for the model and loading plot is used to interpret the relations among variables and clusters observed in the score plot. This first model is good (Figure 2.a) where the separation among the four classes is clearly shown when the plot is in 2D. The total variance of the data explained by the PCA model built was 99% from PC1 (Figure 2.b). In Supplementary Figure 2.C, the plotted PC1 loadings values of the spectral data are shown, representing the regions of the spectra where the differences among sesame oil samples are more evident.



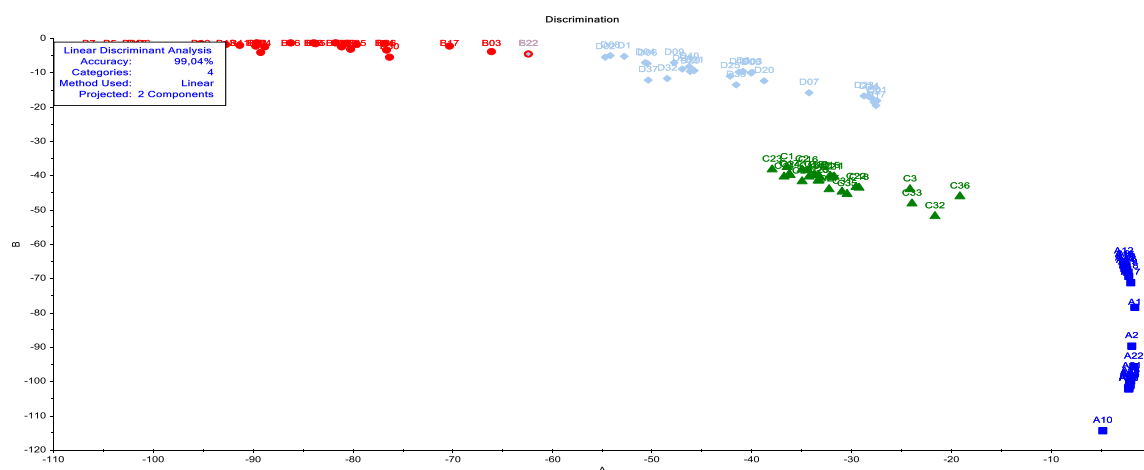
**Figure 2:** (a) PC1 / PC2 Score plot by PCA analysis on the calibration set : A (pur sesame oil) ; B (SO-CO) ; C (SO-SBO) ; D (SO-SFO); (b) Plot of explained variance (%) of PCs used in the PCA model; (c) PC1 loadings plot

The factorial contributions for discriminating the cultivars along the positive axis of PC1 were associated with the spectral windows of 3000–600  $\text{cm}^{-1}$ . Such findings prompted us to perform a more detailed analysis, taking into account the data set related to typical fingerprint regions of carbohydrates (1200–900  $\text{cm}^{-1}$ ), proteins (1680–1540  $\text{cm}^{-1}$ ) and lipids (3000–1700  $\text{cm}^{-1}$ ) to better identify and discriminate the oils samples according to their biochemical

discrepancies. The results from Figure 2.c indicated that the spectral regions associated with lipids was the best ones for oils discrimination by PCA.

### 3.3. PCA-LDA modeling

Since LDA is subject to the constraint that the number of features should not exceed the number of samples, our high-dimensional spectral data were reduced to PCs by PCA prior to LDA. Thus, PCA was applied on the infrared spectra, and the resulting score matrix was used to make the matrix of the predictors. This matrix and the vector of the membership information related to the oils samples were then subdued to LDA procedure. The results obtained showed that the samples were discriminated on the basis of this processing method. In fact, the obtained PCA-LDA model was able to discriminate all samples of the calibration set with a correct classification of 99.04%, with only one sample (B22) simultaneously present in more than one class. This result can be seen in the score plot of Figure 3.



**Figure 3:** Discriminant value plot for classes A and B by calibration set: PCA-LDA application.

This result is confirmed by confusion matrix (Table 1). The confusion matrix is a matrix used for visualization for classification results from supervised method, PCA-LDA. It carries information about the predicted and actual classifications of samples, with each row showing the instances in a predicted class, and each column representing the instances in an actual class. Table.1 shows that all samples are well classified, while one sample of B class resulted classified in D class.

**Table 1:** Confusion matrix of calibration set, carried out by PCA-LDA

Confusion matrix	A	B	C	D
Predicted	1	2	3	4
A	26	0	0	0
B	0	25	0	0
C	0	0	26	0
D	0	1	0	26

### 3.4. Classification of new samples by PCA-LDA model

Discrimination analysis model (PCA-LDA) is trained with calibration samples from four classes and is used to classify future samples (validation set) into one of these classes [48].



**Table 2:** Prediction matrix from application of the PCA-LDA classification model on the test set

Samples	A	B	C	D	Prediction
SA1	-1,575887	-86,51471	-35,56668	-41,58485	A
SA2	-1,568647	-89,98235	-36,34878	-43,87392	A
SA3	-1,678567	-86,11919	-36,27672	-41,42366	A
SA4	-1,529627	-88,19259	-35,5844	-42,64824	A
SA5	-5,149502	-113,5953	-56,56429	-61,78878	A
SA6	-1,613447	-78,4326	-31,14713	-36,01968	A
SA7	-2,613924	-65,89288	-23,21497	-27,54109	A
SA8	-2,404222	-67,42587	-24,44592	-28,57779	A
SA9	-2,560745	-66,23978	-23,61405	-27,78508	A
SA10	-2,010583	-71,34346	-24,71667	-31,00459	A
SA11	-2,314725	-68,35538	-23,74545	-29,08528	A
SA12	-2,505388	-66,64686	-23,85805	-28,05294	A
SA13	-1,726016	-95,81341	-36,27383	-47,64269	A
SA14	-2,594192	-66,79121	-21,94069	-27,99534	A
SB1	-89,55863	-1,451615	-44,57246	-11,615	B
SB2	-84,12794	-1,401732	-40,45201	-9,748106	B
SB3	-61,44745	-3,684931	-25,35872	-3,738296	B
SB4	-62,10003	-3,865659	-24,37315	-3,940195	B
SB5	-89,15907	-4,074146	-57,60746	-14,36884	B
SB6	-91,28149	-2,150882	-51,80747	-13,1825	B
SB7	-92,72222	-1,920801	-50,9644	-13,34638	B
SB8	-70,26964	-2,301967	-30,62302	-5,704131	B
SB9	-102,4026	-2,914503	-58,78368	-17,29649	B
SB10	-62,31729	-4,601485	-22,17997	-4,270819	D
SB11	-76,73108	-1,626932	-38,61776	-7,735665	B
SB12	-86,194	-1,392191	-42,65175	-10,49743	B
SB13	-37,5735	-19,01538	-39,72921	-8,433091	D
SB14	-65,13879	-16,32876	-11,96653	-11,61174	D
SC1	-31,75881	-43,37833	-1,404812	-18,99026	C
SC2	-36,18385	-38,19325	-1,60069	-16,87901	C
SC3	-36,44261	-37,95787	-1,626203	-16,80025	C
Samples	A	B	C	D	Prediction
SC4	-33,46603	-38,86212	-1,456703	-16,5629	C
SC5	-30,80545	-41,70024	-1,390784	-17,6581	C
SC6	-38,55695	-38,15515	-1,793028	-17,51058	C
SC7	-31,6921	-42,54243	-1,391125	-18,43589	C
SC8	-36,42803	-37,55285	-1,6415	-16,5384	C
SC9	-31,68388	-40,44242	-1,396731	-17,0897	C
SC10	-33,71817	-39,8349	-1,438688	-17,25191	C
SC11	-32,15649	-40,18974	-1,402463	-17,05561	C
SC12	-36,281	-39,70105	-1,569912	-17,86769	C
SC13	-34,24426	-40,3311	-1,450163	-17,71219	C
SC14	-34,95502	-41,8214	-1,482215	-18,86047	C
SD1	-47,26333	-7,098797	-20,79443	-1,693485	D
SD2	-61,62014	-4,163665	-23,41014	-3,914517	D
SD3	-27,56329	-19,62702	-25,65405	-4,854594	D
SD4	-52,9746	-7,051202	-17,31579	-2,884196	D
SD5	-56,35751	-4,704831	-23,17022	-2,823231	D
SD6	-56,32062	-4,939747	-22,13201	-2,869534	D
SD7	-58,86789	-4,86975	-21,62968	-3,46454	D
SD8	-47,67154	-6,993454	-20,67339	-1,724898	D
SD9	-54,12932	-7,601542	-16,27483	-3,452816	D
SD10	-51,57137	-7,736026	-16,21868	-2,845567	D
SD11	-53,9338	-7,295388	-16,78017	-3,250038	D
SD12	-28,24631	-17,14407	-14,81885	-2,465916	D
SD13	-41,27499	-9,959327	-16,12819	-1,490372	D
SD14	-38,70907	-12,37109	-12,40438	-2,006287	D

This step allowed to classify the new samples into the classes previously established. According with the LDA procedure, the prediction matrix (Table.2) was carried out from the validation process and showed the discriminated values for each class, as well as the predicted class for each sample. Table.2 shows the classification results with the comparison between the predicted results of each class and the theoretical reference classes. The rate of correct classification was 94.64% within the test set. In particular 53 samples were safely assigned in the four classes, while three samples resulted classified in another class.

## 4. Conclusion

This paper proposes a new method for the qualitative analysis of pure sesame oil (SO) and colza oil (CO), soybean oil (SBO), sunflower oil (SFO) as adulterants in extra sesame oil by applying Attenuated Total Reflectance-Fourier Transform Mid Infrared Spectroscopy (ATR-FTMIR) associated with chemometric techniques. Linear discriminant analysis (LDA) was adopted as classification method, and Principle component analysis (PCA) was employed to compress the original data set into a reduced new set of variables before LDA. On the basis of principal component analysis (PCA), four distinctive clusters were recognized. Then, PCA-LDA was performed to assess the discrimination capacity of the measurement data between the four study oils. Application of PCA-LDA on an external test set of fifty six samples allowed to classify them into four groups with a correct classification of 94.64%. These results indicate that ATR-FTMIR with PCA-LDA can be a useful tool for rapid detection of sesame oil adulteration with seed oils. Its application was rapid and simple because no chemical treatment of samples was required. This approach that would be considered as an effective and feasible method to control the quality of sesame oil.

## References

- [1] S. Esslinger, J. Riedl, C. Fauhl-Hassek. Food Research International. 60 (2014) 189–204.
- [2] A. Tay, R. K. Singh, S. S. Krishnan & J. P. Gore. LWT-Food Science and Technology. 35 (2002) 99–103
- [3] J. H. Kalivas, A. G. Constantinos, M. Marianna, I. Tsafaras, A. P. Eleftherios, A. M. George. Food Chemistry. 148 (2014) 289–293
- [4] E. Anklam & R. Bantaglia. Trends in Food Science and Technology. 12 (2001) 71–102.
- [5] R. Garrido-Delgado, L. Arce, A.V. Guamán, A. Pardo, S. Marco, M. Valcárcel. Talanta. 84 (2011) 471–479.
- [6] M. D. Guillen, & N. Cabo. Journal of the Science of Food and Agriculture. 80 (2000) 2028–2036.
- [7] G. Dobson. European Journal of Lipid Science and Technology. 103 (2001) 815–840.
- [8] Y. W. Lai, E. K. Kemsley & R. H. Wilson. Journal of Agricultural and Food Chemistry. 42 (1994) 1154–1159.
- [9] D. B. Dahlberg, S. M. Lee, S. J. Wenger & J. A. Vargo. Applied Spectroscopy. 51 (1997) 1118–1124.
- [10] N. Dupuy, L. Duponchel, J. P. Huvenne, B. Sombret & P. Legrand. Food Chemistry. 57 (1996) 245–251.
- [11] B. F. Ozen, I. Weiss & L. J. Mauer. Journal of Agricultural and Food Chemistry. 57 (2003) 5871–5876.
- [12] M. Safar, D. Bertrand, P. Robert, M. F. Devaux & C. Genot. Chemists\_ Society. 71 (1994) 371–377.
- [13] M. De Luca, W. Terouzi, G. Ioele, F. Kzaiber, A. Oussama, F. Oliverio, R. Tauler, G. Ragno. Food Chemistry. 124 (2011) 1113–1118.
- [14] G.F. Zagonel, P. Peralta-Zamora, L.P. Ramos. Talanta. 63 (2004) 1021-1025.
- [15] H. Yang, J. Irudayaraj & M. M. Paradkar. Food Chemistry. 93 (2005) 25–32.
- [16] M. De Luca, W. Terouzi, F. Kzaiber, G. Ioele, A. Oussama, G. Ragno. International Journal of Food Science and Technology. 47 (2012) 1286–1292.
- [17] M. Cocchi, C. Durante, A. Marchetti, C. Armanino & M. Casale. Analytica Chimica Acta. 589 (2007) 96-104.
- [18] M. Casale, N. Sinelli, P. Oliveri, V. Di Egidio & S. Lanteri. Talanta. 80 (2010) 1832–1837.
- [19] W. Terouzi, Z. Ait Yacine, F. Kzaiber, A. Boulli, A. Oussama. Kernel olive: International Journal of Chemtech Applications. Vol. 3, Issue 4 (2014) 01-13



- [20] Y. Chen, M. Y. Hie, Y. Yan, S. B. Zhu, S. P. Nie, C. Li et al. *Chimica Acta*. 618 (2008) 121–130.
- [21] B. M. Nicolăi, K. Beullens, E. Bobelyn, A. Peirs, W. Saeys, K. I. Theron et al. *Postharvest Biology and Technology*. 46 (2007) 99–118.
- [22] H. Zhao, B. Guo, Y. Wei, B. Zhang. *Food Chemistry*. 152 (2014) 316–322.
- [23] S. Wold, K. Esbensen & P. Geladi. *Chemom. Intell. Lab. Syst.* 2 (1987) 37.
- [24] B.G.M. Vandeginste, D.L. Massart, L.M.C. Buydens, S.D.E. Jong, P.J. Lewi, J. Smeyers-Verbeke. *Handbook of Chemometrics and Qualimetrics: Part B*, Elsevier, Amsterdam, The Netherlands, 1998, p. 88.
- [25] G. Ivosev, L. Burton & R. Bonner. *Anal. Chem.* 80 (2008) 4933.
- [26] G. Brito, J. Andrade, J. Havel, C. Diaz, F. Garcia, E. Peña-Méndez. *Meat Sci.* 74 (2006) 296–302.
- [27] M. C. M. Ferreira, M. A. Morgano, S. C. N. Queiroz & D. M. B. *Meat Science*. 69 (2000) 259–265.
- [28] D. Granato, I.A. Castro, F.U. Katayama. Assessing the association between phenolic compounds and the antioxidant activity of Brazilian red wines using chemometrics. *LWT-Food Science and Technology*, in press, doi:10.1016/j.lwt.2010.05.031.
- [29] A.M. Martínez, A.C. Kak. *IEEE Trans. Pattern Anal. Mach. Intell.* 23 (2001) 228–233.
- [30] T. Naes, T. Isaksson, T. Fearn & T. A. Davies. *User-friendly guide to multivariate calibration and classification*. NIR Publication. 2002.
- [31] D. Massart. *Uit: Verhandelingen van de Koninklijke Academie voor Geneeskinde van België*, LIX. 1997, 4, 287–325.
- [32] R.M. Alonso-Salces, C. Herrero, A. Barranco, D. Lopez-Marquez, L. Berrueta, F. Gallo, B. Vicente. *Food Chem.* 97 (2006) 438–446.
- [33] S.D. Brown, R. Tauler & B. Walczak. *Comprehensive Chemometrics: Chemical and Biochemical Data Analysis*. 2009, Vol. 2. Elsevier, Amsterdam.
- [34] M. Otto. *Chemometrics Statistics and Computer Application in Analytical Chemistry*, Wiley-VCH, Weinheim, Germany, 1999.
- [35] C. Sârbu, R.D. Naşcu-Briciu, A. Kot-Wasik, S. Gorinstein, A. Wasik & J. Namieśnik. *Food Chemistry*. 130 (2012) 994–1002.
- [36] J. Yang & J. Yu Yang. *Pattern Recogn.* 36 (2003) 563–566.
- [37] S. Roussel, W. Bellon-Maurel, J. M. Roger, P. Grenier. *Food Eng.* 60 (2003) 407–419.
- [38] N. Dupuy, O. Galtier, D. Ollivier, P. Vanloot and J. Artaud. *Analytica Chimica Acta*. 666 (2010) 23–31.
- [39] A. Rohman & Y.B. Che Man. *Food Res. Int.* 43 (2010) 886–892.
- [40] M. Lopez-Sanchez, M.J. Ayora-Canada, A. Molina-Diaz. *Food Chem.* 58 (2010) 82–87.
- [41] M. D. Guillen & N. Cabo. *Journal of the American Oil Chemists Society*. 74 (1997) 1281–1286.
- [42] M. J. Lerma-Garcia, G. Ramis-Ramos, J. M. Herrero-Martinez & E. F. Simo-Alfonso. *Food Chemistry*. 118 (2010) 78–83.
- [43] F. A. Iñón, J. M. Garrigues, S. Garrigues, A. Molina and M. de la Guardia. *Analytica Chimica Acta*. 489 (2003) 59–75.
- [44] A. A. Christy, S. Kasemsumran, Y. Du & K. Ozaki. *Analytical Sciences*. 20 (2004) 935–940.
- [45] A. Savitzky & M.J.E. Golay. *Analytical Chemistry*. 36 (1964) 1627–1639.
- [46] A. Hirri, M. Bassbasi & A. Oussama. *International Journal of Advanced Technology & Engineering Research*. 3 (2013) 59–62.
- [47] K.H. Esbensen. *Multivariate data analysis-in practice. An introduction to multivariate data analysis and experimental desing*. Oslo: CAMO. 2000.
- [48] X. Lu, Y. Zi-Hong, Y. Si-Min, S. Peng-Tao, C. Hai-Feng, F. Xian-Shu, Y. Xiao-Ping. *Analytica Chimica Acta*. 754 (2012) 31–38