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The chemometrics approach applied to FTIR spectral data for the oxidation study of Algerian extra virgin olive oil

[1, 2*]Selaimia, R., [1]Oumeddour, R. and [1]Nigri, S.

[1]*Industrial Analysis Laboratory and Materials Engineering, University 8 May, 1945. B.P.401, Guelma, Algeria*

[2]*Department of Petrochemical and Engineering Processes, Faculty of Technology, University 20 August 1955, Skikda, Algeria*

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Abstract

Thermal oxidative stability study was carried out on four different samples of extra virgin olive oil. Physicochemical methods were used for the determination of indices and Fourier transform infrared spectra were employed for differentiation and classification of olive oil produced in Algeria. In order to determine the thermal effect on oxidation, the samples were subjected to heating treatment at 60°C and evaluated during 90 days using Fourier transform infrared spectra absorption. The spectral regions where the variations were observed have been chosen for developing partial least square (PLS) model. Four and six factors obtained by cross validation were sufficient to monitor the oxidation process. Once the oxidation process committed under the influence of time during 90 days and heated at 60°C, a multitude of molecules forming a dynamic complex system appeared.

Introduction

Extra virgin olive oil (EVOO) is widely known because of its high health benefits and sensory quality in comparison to other oils and fats (Bendini *et al*., 2007). Olive culture has a great economic and social importance in the Mediterranean area and moreover the olive oil is representative of the culture and alimentary customs of this region.

Olive oil is a combination of different components, each of which plays an important role in its characterization.

Characterization can be done in different ways, but only a global assessment of the analytical parameters can assure an objective evaluation. This approach is also essential to follow the possible changes over time and to light out possible alteration or adulteration. Virgin olive oil, due to its use without refining, shows very interesting nutritional and sensorial properties, being one of the pillars of the so called Mediterranean diet (Aguilera *et al*., 2005). Compared to other vegetable oils: tomato seed oil (Kim *et al*., 2010; Giuffrè *et al*., 2016a; Giuffrè *et al*., 2016b), 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 (COI, 2015).

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 Council states that FFA in extra virgin olive oil should be ≤ 0.8%, while the FFA content in lower-grade olive oil could be as high as 2% for virgin olive oil and 3.3% for ordinary virgin olive oil (COI, 2015). The oxidative stability of oils could be defined as their resistance to oxidation. This is an important indicator of performance and shelf-life, and depends on the composition of the sample and on the conditions to which it is subjected (Guillèn and Cabo, 2002).

The oxidative stability, sensory quality and health properties of virgin olive oil stem from a prominent and well-balanced chemical composition (Servili *et al*., 2004). Oxidation of edible oils is influenced by an energy input such as light or heat, composition of fatty acids, types of oxygen, and by compounds such as metals, pigments, phospholipids, free fatty acids, mono and diacylglycerols, thermally oxidized compounds, and antioxidants. Many efforts have been made to improve the oxidative stabilities of oils by systematic studies on the effects of these factors (Choe and Min, 2006).

Infrared spectroscopy has scarcely been used for the study of fats and oils. However, even with the dispersive technique, determination of the degree of unsaturation of fats and oils from the intensity

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*Corresponding author.
Email: *selaimia_radia2000@yahoo.fr*
Tel: + 213 (0)5 54 68 70 53

of concrete infrared spectroscopic bands has been known for a long time (Sinclair *et al*., 1952; Arnold and Hartung 1971; Muniategui *et al*., 1992; Maggio *et al*., 2009; Lerma-Garcíaa *et al*., 2011).

Over the past 15 years, as a result of the development of FTIR spectroscopic instrumentation, the application of this technique expanded in food research and particularly has become a powerful analytical tool in the study of edible oils and fats. FTIR spectroscopy is a rapid, non-destructive technique with minimum sample preparation necessary. It allows the qualitative determination of organic compounds as the characteristic vibrational mode of each molecular group causes the appearance of bands in the infrared spectrum at a specific frequency, which is further influenced by the surrounding functional groups (Vlachos *et al*., 2006). FTIR is also an excellent tool for quantitative analysis since the intensities of the spectral bands are proportional to concentration.

Moreover, the changes observed in the frequency data of some bands and also in the ratios of absorbances of the FTIR spectra during the oxidation process have been studied (Van de Voort *et al*., 1994; Russin and van de Voort, 2004). FTIR has been also used to distinguish extra virgin olive oils (EVOOs) from different geographical origins (Tapp *et al*., 2003; Bendini et al., 2007; Galtier *et al*., 2007) and different genetic varieties (Gurdeniz *et al*., 2007).

FTIR applications addressed to detect olive oil adulteration with low cost edible oils (Ozen and Mauer, 2002; Tay *et al*., 2002; Baeten *et al*., 2005), to evaluate olive oil freshness (Sinelli *et al*., 2007), to study changes produced by frying (Valdes and Garcia, 2006) and to assess oil oxidation (Guillèn and Cabo, 2002; Muik *et al*., 2007) have also been described.

In the present study, FTIR spectroscopy is used for characterization of four olive oil grown in northern Algeria and also for the monitoring of the oxidation process. Volatile profile of Algerian olive oils was established using headspace solid phase micro-extraction (HS-SPME) and gas chromatography coupled to mass spectrometry in previous work of Nigri *et al*. (2012) and Louadj and Giuffrè (2010). Initially, an evaluation of a typical FTIR spectrum of an olive oil samples was presented.

In addition, the combination of chemometric techniques with the FTIR spectroscopy was used, to discriminate olive oils from different origin and/or extracted according to different processes. The spectral regions where the variations were observed were chosen for developing PLS, This approach will be also tested to determine authenticity of olive oils.

Materials and Methods

Samples collection

Four extra virgin olive oil (EVOO) samples from different East Algerien regions of Guelma (HG), Collo (HC), Milla (HM) and Skikda (HS) were harvested in the fall of 2012. Olive fruits were manually harvested and extra-virgin olive oil was extracted with a modern system with metal crusher and centrifugation of the olive paste. All samples analysed using official methods provided in European Regulation.

For the monitoring of the oxidation process, four vials containing 50 ml of each extra virgin olive sample were placed in stove set at 60°C. Finally, at different times of incubation (every 10 days) during 90 days, 5 ml for each oil sample was taken for measurements. All heated samples were cooled at room temperature (24 ±1°C) for 90 min and stored in bottles at 10°C before FTIR analyses.

Physical and chemical parameters determination

The free fatty acid, the peroxide value, saponification number and iodine value were determined according to European Union standard methods (COI, 2015).

Free fatty acid

Free fatty acid (FFA) was determined by titration of an oil solution dissolved in ethanol/ether (1:1, v/v) with 0.1 M potassium hydroxide ethanolic solution, and expressed as percent of oleic acid. The FFA was determined according to ISO 660 method.

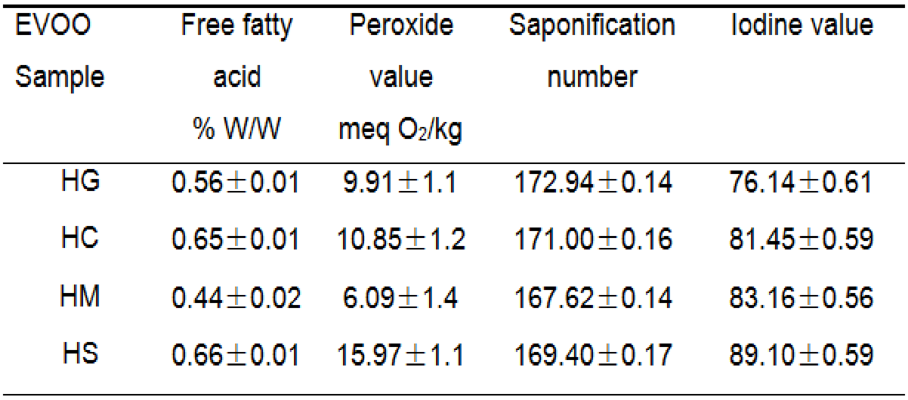
Peroxide value

Peroxide value (PV), given in milliequivalents of active oxygen per kilogram of oil (meq/kg of oil), was determined as follows: a mixture of oil and chloroform/acetic acid 3:2 (v/v) was left to react in dark with saturated potassium iodine solution; the free iodine was then titrated with a sodium thiosulphate solution. The PV was determined according to ISO 3960 method.

Saponification number

The saponification number (SN) is a measure of the average molecular weight of the triacylglycerols in a sample. The oil sample is saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali required for saponification is determined by titration of the excess potassium hydroxide with standard hydrochloric acid. The SN was determined according to ISO 3657 method.

Table 1. Physicochemical properties of HG, HC, HM and HS samples before the thermal treatment



EVOO Sample	Free fatty acid % W/W	Peroxide value meq O_2/kg	Saponification number	Iodine value
HG	0.56±0.01	9.91±1.1	172.94±0.14	76.14±0.61
HC	0.65±0.01	10.85±1.2	171.00±0.16	81.45±0.59
HM	0.44±0.02	6.09±1.4	167.62±0.14	83.16±0.56
HS	0.66±0.01	15.97±1.1	169.40±0.17	89.10±0.59

Iodine value

The iodine value (IV) gives a measure of the average degree of unsaturation of a lipid. The oil sample taken in carbon tetrachloride is treated with a known excess of iodine monochloride solution in glacial acetic (Wijs solution). The excess of iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution. The IV was determined according to ISO 3961 method.

FTIR spectra acquisition

A Perkin-Elmer Spectrum one FTIR spectrophotometer equipped with a deuterated triglycerine sulphate (DTGS) detector was used to collect FTIR spectra with a resolution of 4 cm^{-1} at 20 scans. A small quantity (2 µL) of the sample was deposited using a Pasteur pipette between two well-polished KBr disks, creating a thin film. Duplicate spectra were collected for the same sample. All spectra were recorded from 4000 to 500 cm^{-1} and processed with the computer software program Spectrum for Windows (Perkin-Elmer).

Chemometrics analysis

Principal component analysis of FTIR spectra using transmittance at 16 wavenumber regions was carried out using the Minitab 16 version. The frequency regions of 3480 to 2670 cm^{-1} and 1750 to 720 cm^{-1} were picked up to monitor oxidation EVOO. The appropriate number of PLS-factors is determined by application of Haaland and Thomas's criterion (1988), based on the minimal stable predicted residual sum of squares PRESS. The performance of the model was evaluated by the coefficient of determination R^2 and the root mean square error of cross validation RMSECV.

$$RMSECV_k = \sqrt{\frac{PRESS_K}{n}}$$

Where: k is the number of latent variables; $PRESS_k$ is the sum of squared prediction error for the model and n is the number of calibration samples (De la Mata *et al.*, 2012).

Results and Discussion

Analytical results

The analytical results for the four studied samples are summarised in Table 1. Over all measurements were performed three times for each sample. As shown in Table 1, in all samples examined, the free fatty acid content was much lower than the upper limit of 0.8% established for the best commercial quality olive oil, designated as "extra" virgin (COI, 2015). The peroxide value of the oils analyzed was below the limit of 20 meq of oxygen/kg of oil, which is accepted as the limit for extra virgin olive oil (COI, 2015).

Evaluation of an FTIR spectrum

In order to obtain information on the structural changes involved in the oxidative process after heat treatment, we have studied the FTIR spectra of four different extra-virgin olive oil samples at T = 25°C. All the four native samples (before thermal treatment) showed the same profile, the spectrum was characterized by the presence of several peaks in the explored wavenumber range.

In the Figure 1 it is shown the comparison between olive oil heated at 60°C during 0 to 90 days. There were two interesting spectral regions from 3480 to 2670 cm^{-1}, where the activity of stretching vibrations of fatty acids and hydroperoxides was observed and from 1750 to 720 cm^{-1}, where the vibrational activity of conjugated bonds and of bending vibrations of aliphatic compounds were observed.

It is well known that after heat treatment some of peculiar infrared bands of an olive oil sample are modified (Guillèn and Cabo, 2000). The initial

Table 2. Statistical parameters curried out by full cross-validation on PLS

Sample	Optimum number of PLS factors	R^2 Calculated	R^2 predicted	RMSECV	F	P	S
HM	4	0.9928	0.9022	8.48	103.55	0.002	14.11
HS	6	1.000	0.8997	7.90	7304.39	0.030	0.012
HC	4	0.988	0.7895	11.98	41.92	0.023	28.12
HG	6	0.998	0.8951	9.30	282.9	0.000	4.85

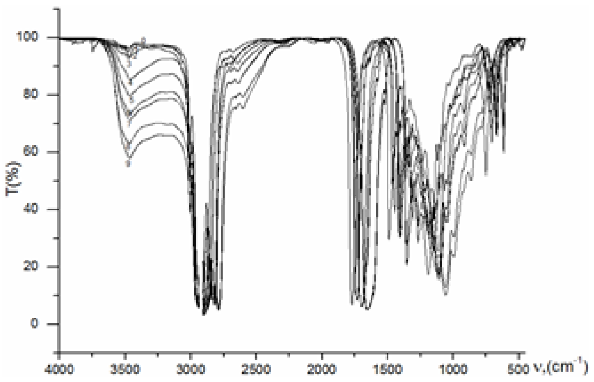


Figure 1. FTIR spectra of olive oil sample (HM) after 90 days at 60°C

phase of the oxidative process involves the increase of the hydroperoxide content and of some secondary oxidation compound; next steps are characterized by the breaking of the ester bonds between the fatty acids and glycerol, that decreases the unsaturation degree and increases the trans isomers concentration (Navarra *et al.*, 2011). We can consider, the band at 3474 cm^{-1} as a fingerprint of the oxidation process associated to the C–H stretching vibration of the cis-double bond (CH).

The band position remains almost unaltered or undergoes a very low shifting towards higher wavenumber during the first time of oxidation (Guillèn and Cabo, 2000). However, a shift towards lower wavenumbers and the decrease of its amplitude may be a sign of advanced oxidation, revealed up to complete quenching at very advanced stages of the oxidation.

These effects can be attributed to the disappearance of cis double bonds, as well as isomerization of cis to trans groups, alongside with hydroperoxide generation. For this reason frequency and absorbance of band near 3006 cm^{-1} suffer changes as the oxidation process advances (Guillèn and Cabo, 2002). The shifting extent is dependent on the oil typology. The oxidative behaviors of the four oils from different geographical regions were not the same.

The analysis of the variations in FTIR spectra was challenging, because these changes were very weak and there were so many peaks and shoulders without resolution, for a better visualisation of trends, the compression of the spectral information is needed. Principal component analysis (PCA) is one of the most used tools for this kind of works. (Maggio *et al.*, 2011).

Infrared spectroscopic techniques coupled to chemometric classification methods is a powerful tool for the traceability of olive oil samples with designated origin (Bevilacqua *et al.*, 2012). The spectral region where the variation was observed was chosen for developing model.

Chemometrics analysis

In the PLS calibration models, the evaluation of the method linearity was carried out in order to show a proportional relationship between responses versus analyte concentrations. This technique allows us to build predictive models using a large number of data characterized by strong collinearity (Ragni *et al.*, 2012). In the optimization of frequency regions used containing the significant information were selected and all the useless information coming from the interferences or instrumental drigs were ignored.

Using PLS, the excellent model was obtained for HS sample as shown in Table 2, the R^2 value obtained was 1.000 (in calibration) and 0.89997 (in prediction) with lowest RMECV (7.90). Six latent factors were selected for building PLS models. PLS offers a better calibration model for HG than HM. Inversely it gives better prediction model for HM. The high value of RMECV (11.98) was obtained for Collo EVOO (HC).

Figure 2 shows the scatter plots for the relationship

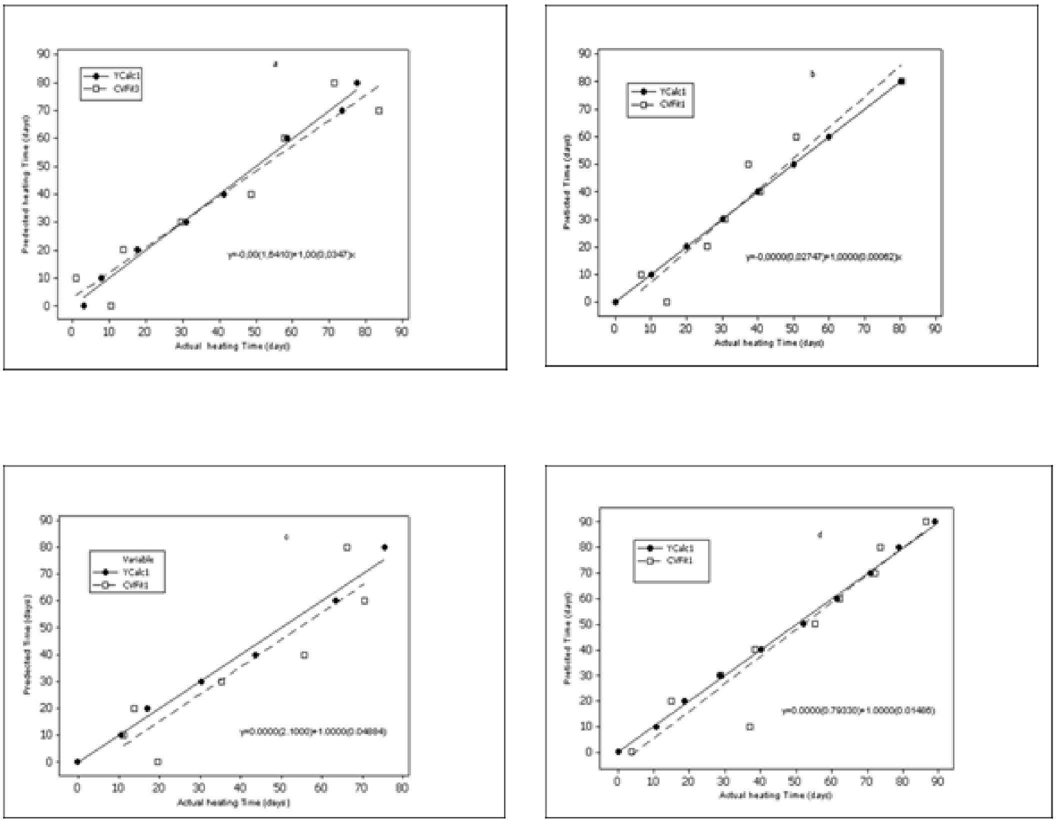


Figure 2. Correlation plot of the actual versus and the predicted heating time: a) HM-b) HS-c) HC- d) HG

between actual and FTIR predicted heating time for all EVOO. It indicates a close relationship between two variables assessed using FTIR-PLS, the relationship between actual and predicted heating time shows a very good correlation with R^2 of 0.993, 1.0000, 0.988 and 0.998 for HM (a), HS (b) , HC (c) and HG (d) respectively.

Principal component analysis

PCA allows visualizing the information data set in a few principal components retaining the maximum possible variability within that set. When a PCA procedure is used, the amount of original variables is reduced to a few latent variables or principal components (PCs) (De Luca *et al.*, 2011). Figure 3 shows the score plot of the PC1 vs. PC2 principal components of the PCA model performed on the olive oil samples. The score for all samples in two first principal components that explain 93.3% of the data matrix variance is depicted in Figure 3. It clearly shown that olive oil samples are sufficiently separated in three groups: Highly, moderately and weakly oxidized samples. Moreover, it is shown that EVOO have positive scores in the first principal component (PC1), while those exposed for 40 to 60 days and for 70 to 90 days exhibit negative PC1 value. The PC2, on the other hand, seems to classify the samples exposed for 40 to 90 days in two groups, samples exposed at 60°C for 40 to 60 days were characterized by positive PC2 value while those exposed for 70 to 90 days by negative PC2 value.

The importance of the contribution into PC1 of the FTIR spectra with transmittance at 1119, 1096, 1239, 2854, 1377, 1418, 1163, 1656, 1746, 3007, 3474 and 723 cm^{-1} were evident from the corresponding loading plot. The wavenumber which correspond to 3474 cm^{-1} was attributed to (O-H), 3007 cm^{-1} was ascribed to C-H stretching vibration of the cis-double bond (=CH), 1746 cm^{-1} was ascribed to –(C=O) group and 1656 cm^{-1} was related to (C=C) stretching vibration of cis-olefins, 1418 cm^{-1} was assigned to Rocking vibrations of CH bonds of cis-dissubstituted olefins. 1239 cm^{-1} was attributed to (C=C) and 1163, 1119, 1096 cm^{-1} were due to –C-O (ester) vibrations. The results obtained by PCA (models show) very good discrimination between heated olive oil samples.

Conclusion

The effects of the oxidation process induced by thermal treatment on some extra-virgin olive oil samples from different region were investigated by FTIR spectroscopy combined to chemometric analysis. The IR spectra show that the oxidation process starts with the formation of hydroperoxides. These unstable molecules were degraded, forming

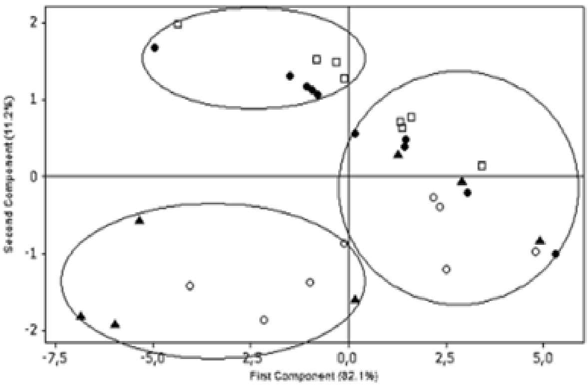


Figure 3. Exhibited the PCA score plot obtained from the correlation matrix of peak transmittance at 17 frequency regions ▲ (HC), ○ (HM), ● (HG), □ (HS)

alcohols, aldehydes and ketones as highlighted by the modification of the spectral component. Within the observation time (90 days), the most significant regions included the band related to 3474 cm^{-1} attributed to (O-H), 3007 cm^{-1} ascribed to C-H stretching vibration of the cis-double bond (=CH),1746 cm^{-1}ascribed to –(C=O) group and 1656cm^{-1} (C=C) stretching vibration of cis-olefins, 1418 cm^{-1} assigned to rocking vibrations of CH bonds of cis-disubstituted olefins. 1239, attributed to (C=C) and 1163, 1119, 1096 cm^{-1} caused by –C-O (ester). FTIR-PLS models were able to distinguish the different oils' regions quality and the effect of variable heating times on oils quality. This approach could be useful for monitoring the oxidative status of heating oil's. Once the oxidation process committed under the influence of time and heat, multitude of molecules forming a dynamic complex system appeared.

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