

## Detection of thermal processing impact on olive and sunflower oil quality by FTIR spectroscopy

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Received: 19 January 2013; Accepted: 27 February 2013

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### Abstract

The main goal of this study was to assess the effectiveness of FTIR spectroscopy to detect the changes occurred in quality of olive oil (OO), sunflower oil (SFO) and their mixture as effect of convective heating for 8 and 16 h under frying procedures. The IR bands changing during heating in oil samples have been examined. It can be claimed that a band shift noticed at 3011 cm<sup>-1</sup> assigned to the C-H stretching vibration of the cis-double bond allows estimating both OO adulteration by SFO addition as well as the extent of oxidative degradation occurred in oils as a result of heating at high temperature. The changes reported in the spectral domains 2800-3050 cm<sup>-1</sup>, 1600-1800 cm<sup>-1</sup> as well as 700-1200 cm<sup>-1</sup> after heating at elevated temperature aid to the monitoring of oxidation process. Based on calibration curves absorbance at some wave numbers (the most relevant seem to be 3011, 3006 and 912 cm<sup>-1</sup>) versus percent of SFO added in OO it is possible to determine the degree of OO adulteration. A significant positive correlation (R>0.95) were found for untreated oil samples. In addition, these curves could be useful to estimate the extent of oxidative alteration that occurred in oils as effect of heating. The main drawback is the fact that with extend of heating time the linearity of these curves decreased. After 16 hours of thermal treatment these curves are not useful to assess the degree of OO adulteration by SFO addition. Data achieved by carrying out of this study highlighted that FTIR spectroscopy showed a high potential to be a useful method for monitoring the process of oxidative degradation occurred in edible oils subjected to heating at high temperature.

**Keywords:** Fourier transform infrared (FTIR) spectroscopy, olive oil, sunflower oil, adulteration, convective heating, thermo-oxidative degradation.

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### 1. Introduction

The oil stability during heating is an important parameter for ensuring that oils show a good resistance at elevated temperature. During thermal treatments at high temperature, vegetable oils are exposed to oxygen and moisture resulting in different chemical reactions, such as thermo-oxidation, polymerization and hydrolysis. These reactions have as effect the alteration of oil quality. **Frying, by using** of edible oils as cooking

medium, is a method often applied in food industry that involves the maintaining of oil at a high level of temperature, in the range 170-220°C. Sunflower and olive oils are widely used in food processing as source of fatty essential acids [1-3].

OO is especially rich in mono-unsaturated fatty acids (MUFA) like oleic acid (18:1) and palmitoleic acid (16:1) that help to lower LDL or "bad cholesterol" and increase HDL or "good cholesterol" in the blood.

SFO is rich in polyunsaturated fat containing some fatty acids that our bodies cannot produce and thus this kind of oil is an essential part of our diet. These oils contain triglycerides with fatty acids that are different by length of chain, position of double bond in the carbon chain, degree of saturation, etc. The most abundant monounsaturated fatty acid in these oils is the oleic [4-7]. The biggest plus of OO is that it is richer in oleic acid than SFO. It was proved that oleic acid is more stable towards oxidative degradation both at ambient temperatures and during thermal applications that involved high temperatures as cooking and frying [4,8]. Previous researches emphasize that edible oils with high content of oleic acid are less exposed to oxidative degradation during heating than oils that contain high amounts of polyunsaturated fatty acids [9-11]. The changes recorded in oils quality during frying have been previously reported [8-12]. Oxidation of unsaturated fatty acids is one of the major reasons in the development of off-flavor compounds and in the losing of nutritional value of edible oil as well as of processed products resulted after frying [11]. Although many instrumental techniques have been reported as adequate for monitoring of these changes, there are a few numbers of studies that investigated the potential of FTIR spectroscopy for detecting the heating impact on quality of pure and adulterated OO [12-16].

In this study the changes occurred in IR spectra during convective heating at high temperature of OO have been examined in relationship with the content of SFO added as adulteration agent. Some frequencies are selected to be useful for assessing the impact of heating on oils quality because could provide valuable information regarding the extent of thermo-oxidative degradation.

## 2. Materials and methods

**Oil samples.** In this study were used followed oil samples: olive oil - OO (Extra Virgin Olive Oil Minerva, Aceites Borges Pont, Spain), sunflower oil - SFO (Bunica International SA Slobozia) and their mixture obtained by deliberate addition of SFO in established proportions in the range 5 to 50% (v/v) from 5 to 5% to OO. These mixtures were strongly manually shaken for homogenization.

**Heating process in convective oven.** Oil samples were heated under simulated frying conditions

specific to food industry using a convection electric oven.

Experimental design of heating process was developed in agreement with previously studies [1,2].

Preliminary tests were carried out in order to know the thermal regime of this treatment. Oil samples (25.0±0.5g) weighed into the Pyrex Petri dish with inner diameter 11 cm were placed in electrical oven (Esmach, Italy, 1200W, 50Hz) regulated at 200°C and heated for 8 and 16 h. After each heating time the samples were taken out of the oven, cooled and stored at -18°C till the analysis. The **temperatures** of samples from time to time were **determined** by inserting a calibrated chromel-alumel thermocouple (HI 935009, **Hanna Instruments**) into the oil. In Table 1 is shown the internal temperatures of oil during heating as average of values recorded for OO, SFO and their mixture.

**Table 1.** Thermal regime of oil samples during convective heating

Heating in convective oven	Temperature (°C)				
	0	2h	4 h	8h	16h
	23.2	188.6	190.2	191.4	193.2

**FTIR analysis.** A FTIR-8400S Fourier Transform Infrared Spectrophotometer (**Shimadzu**, Japan) equipped with attenuated total reflectance (ATR) accessory was used to obtain the FT-IR spectra. BKG scan and samples were sequentially measured at room temperature in the range 600-4000 cm<sup>-1</sup> with scanning time 60 s and 4 cm<sup>-1</sup> resolution. All spectra were processed with the computer software program Spectrum for Windows XP Professional (Shimadzu). Simple linear regression analysis was applied using the Origin 6.0 software program for obtaining of some correlations between absorbance at specific wave numbers and percent of SFO added in OO for both untreated and thermally processed oil samples.

## 3. Results and discussions

FTIR spectra of investigated oil samples present a lot of bands with different intensities and forms. According to previous study on this topic conducted by Alexa *et al.* [5], based on the absorbance recorded at different wave numbers we could split the FTIR spectra in six different regions in the range 4000 to 600 cm<sup>-1</sup> as follows: *region A* (4000-3100 cm<sup>-1</sup>), *region B* (3100-2800 cm<sup>-1</sup>), *region C* (1800-1600 cm<sup>-1</sup>), *region D* (1600-1390 cm<sup>-1</sup>), *region E* (1390-1200 cm<sup>-1</sup>) and *region F* (1200-700 cm<sup>-1</sup>).

In Figure 1 the FTIR spectra region for OO, SFO and their mixture OO+50%SFO are comparatively shown for untreated samples as well as for oil samples after 8 and 16 h of convective heating.

In the spectral interval ranged from 4000 to 3100  $\text{cm}^{-1}$  the investigated oil samples showed no infrared absorption.

At a closer look to the Figure 1 it is noticed that there are visual differences in the absorbance values registered for untreated and thermally processed oil samples. In the range 3100-2800  $\text{cm}^{-1}$ , assigned to spectral region B, appear the absorption bands near the frequencies 2965, 2935, 2895 and 2855  $\text{cm}^{-1}$ . These bands are characteristic to symmetric and asymmetric vibrations  $\nu(\text{C-H})$ , of the  $\text{CH}_3$  and  $\text{CH}_2$  groupings from aliphatic groupings of alkyl rest from triglycerides which are found in large amounts in vegetable oils. This region also contains a characteristic area in the range 2990-3010  $\text{cm}^{-1}$  which corresponds to the unsaturated aliphatic compounds. The absorption close to 3010  $\text{cm}^{-1}$  represents an index used for detecting the degree of unsaturation of the oils. By quantifying the intensity of absorption near 3010  $\text{cm}^{-1}$  for pure OO, SFO and their mixture, in native state and after heating it is possible to assess their quality. Increasing of the content of SFO in OO had a result the increase of the absorption intensity at 3011  $\text{cm}^{-1}$ , due to the increasing of linoleic acid content in triglycerides composition [5]. From Table 1 it can be noticed that for untreated oil samples the increasing of SFO content in OO induced an increase of absorbance at 3011  $\text{cm}^{-1}$  due to increasing of linoleic acid content in triglycerides composition. Based on calibration curve (absorbance at 3011  $\text{cm}^{-1}$  versus percent of SFO added in OO) it is possible to determine the degree of OO adulteration by SFO addition. A significant positive correlation was found ( $R=0.98$ ). The FT-IR spectra for oil samples show that there are differences in the spectral data recorded close to 3006  $\text{cm}^{-1}$ . According to our result recorded at this frequency there was obtained a high linear correlation absorbance versus percent of SFO added to OO for untreated oil samples, Figure 3. This means that the absorption at this frequency is also used in order to assess the OO adulteration by SFO addition especially for oil samples in initial status.

The spectral data at 3011  $\text{cm}^{-1}$  were not significant changed after 8 h of thermal treatment but with extent of heating time up to 16 h we have come to notice a great decreasing recorded in absorbance values for SFO and mixture OO+50%SFO. This finding pointed out that the OO is more stable than SFO during heating. In addition, thermal instability due to oxidative degradation for the mixture OO+50%SFO has grown worse with the prolonging of thermal treatment. The same findings have kept also at 3006  $\text{cm}^{-1}$ . FTIR spectroscopy can be used to follow the course of oil oxidation but with the increasing of heating time the assessing of OO adulteration by addition of SFO based on the values of absorbance at 3011 and 3006  $\text{cm}^{-1}$  is no possible. Thus, for thermal applications used in food industry, the processors could replace the pure OO with the other one, cheaper such as SFO without to be possible a strict evidence of this fraud.

In the spectral region C we could notice two important bands near 1750 and 1660  $\text{cm}^{-1}$ . Absorption at 1750  $\text{cm}^{-1}$  it is characterized through a band corresponding to the valence vibration of the carbonyl bond of ester  $\nu(\text{C=O})$  and does not bring information about the characterization of oils. The band close to 1660  $\text{cm}^{-1}$  wavelength it is characteristic to the vibration  $\nu(\text{C=C})$  given by the olefinic molecules. The absorption close to 1650  $\text{cm}^{-1}$  provides information regarding also the nature of the double bonds: isolated or conjugated. The isolated double bonds ( $\text{C=C}$ ) show absorption at 1650  $\text{cm}^{-1}$ , conjugated dienes at 1630  $\text{cm}^{-1}$  and conjugated trienes at 1610  $\text{cm}^{-1}$ . For untreated oil samples, we can point that for the absorption at 1753  $\text{cm}^{-1}$ , it was registered significant changes with the increasing of SFO content added in OO due to the fact that SFO is richer in saturated acids than OO. We rely on previous researches conducted by Alexa *et al.* [5] and Vlachos *et al.* [17], which pointed that the absorbance at 1753  $\text{cm}^{-1}$  is characteristic to the oils with a high saturated fatty acids content and short carbohydrate chain. Also, for untreated oils, the absorption intensity close to 1660  $\text{cm}^{-1}$  increased with increasing the SFO percent added in OO. This band corresponds to the double  $\text{C=C}$  bonds and could be correlated with the content of polyunsaturated fatty acids from oil samples.

In spectral region D appeared one absorption band near 1440-1445  $\text{cm}^{-1}$  which correspond to the vibrations of deformation  $\delta(\text{CH})$ .

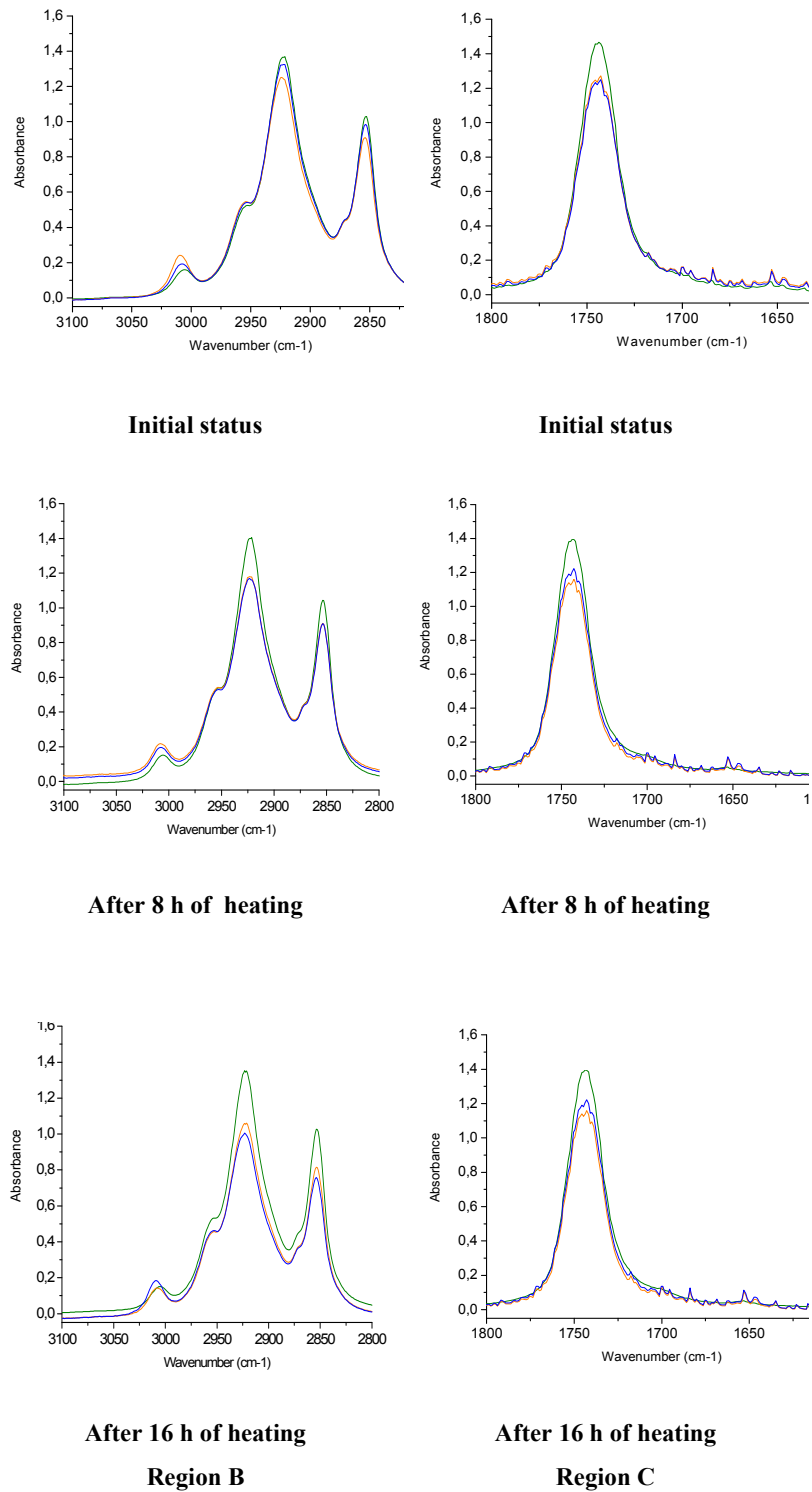


Figure 1. FTIR spectra for pure OO (—), pure SFO (—) and 50%OO+50%SFO (—)

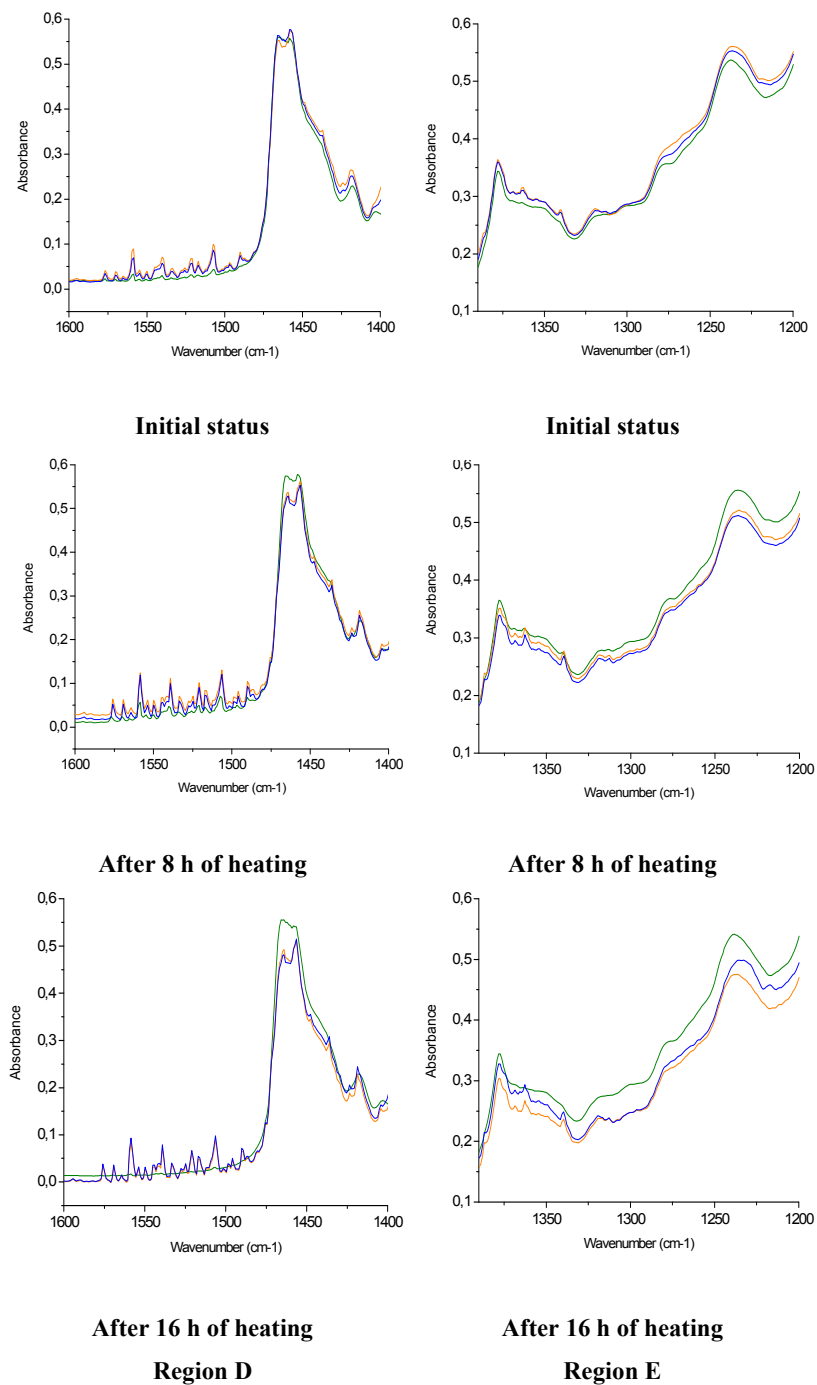
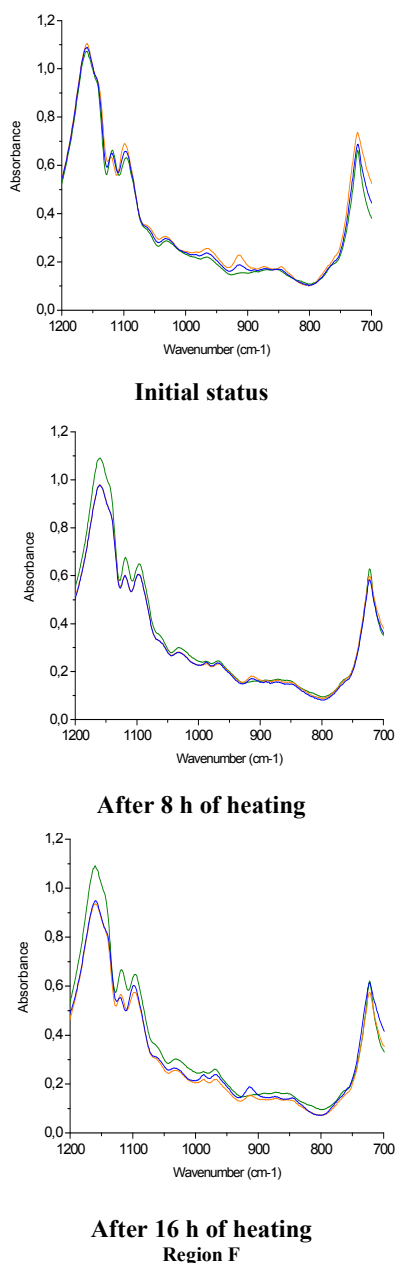


Figure 1. (continued) FTIR spectra for pure OO (—), pure SFO (—) and 50%OO+50%SFO (—)

**Table 2.** Absorbance values recorded in IR for OO, SFO and their mixture in initial status and after convective heating for 8 and 16h

Sample	Absorbance recorded at wave numbers:				
	3011cm <sup>-1</sup>	3006 cm <sup>-1</sup>	1654 cm <sup>-1</sup>	912 cm <sup>-1</sup>	722 cm <sup>-1</sup>
<b>Initial status</b>					
pure OO	0.1360	0.1585	0.0626	0.1547	0.6619
OO+5%SFO	0.1409	0.1653	0.0691	0.1546	0.6534
OO+10%SFO	0.1442	0.1598	0.0689	0.1592	0.6613
OO+15%SFO	0.1462	0.1636	0.0775	0.1632	0.6522
OO+20%SFO	0.1520	0.1678	0.0784	0.1641	0.6578
OO+25%SFO	0.1524	0.1666	0.0799	0.1700	0.6657
OO+30%SFO	0.1582	0.1707	0.0826	0.1746	0.6706
OO+35%SFO	0.1587	0.1697	0.0807	0.1744	0.6654
OO+40%SFO	0.1771	0.1865	0.0902	0.1844	0.6850
OO+45%SFO	0.1810	0.1895	0.0946	0.1876	0.6855
OO+50%SFO	0.1941	0.2027	0.1004	0.1960	0.6943
pure SFO	0.2347	0.2261	0.1152	0.2274	0.7369
<b>After 8 h of heating</b>					
pure OO	0.1279	0.1510	0.0750	0.1599	0.6290
OO+5%SFO	0.1332	0.1538	0.0788	0.1612	0.6351
OO+10%SFO	0.1369	0.1566	0.0812	0.1641	0.6382
OO+15%SFO	0.1410	0.1587	0.0862	0.1669	0.6394
OO+20%SFO	0.1404	0.1575	0.1017	0.1731	0.6327
OO+25%SFO	0.1432	0.1582	0.0981	0.1751	0.6400
OO+30%SFO	0.1531	0.1671	0.1053	0.1840	0.6429
OO+35%SFO	0.1507	0.1632	0.1058	0.1839	0.6509
OO+40%SFO	0.1717	0.1819	0.1133	0.1856	0.6503
OO+45%SFO	0.1843	0.1938	0.0816	0.1705	0.5829
OO+50%SFO	0.2076	0.2143	0.0915	0.1803	0.5973
pure SFO	0.2350	0.2285	0.0908	0.2035	0.6270
<b>After 16 h of heating</b>					
pure OO	0.1259	0.1498	0.0499	0.1544	0.6221
OO+5%SFO	0.1240	0.1448	0.0577	0.1498	0.6308
OO+10%SFO	0.1207	0.1404	0.0586	0.1538	0.6344
OO+15%SFO	0.0921	0.1101	0.0647	0.1624	0.6201
OO+20%SFO	0.0779	0.0944	0.0620	0.1610	0.6216
OO+25%SFO	0.1371	0.1582	0.0668	0.1731	0.5666
OO+30%SFO	0.1210	0.1343	0.0473	0.1499	0.5540
OO+35%SFO	0.1063	0.1179	0.0484	0.1520	0.5455
OO+40%SFO	0.1222	0.1320	0.0505	0.1495	0.5690
OO+45%SFO	0.1405	0.1507	0.0782	0.1800	0.5811
OO+50%SFO	0.1326	0.1398	0.0528	0.1527	0.5748
pure SFO	0.1773	0.1717	0.0643	0.1869	0.6132



**Figure 1 (continued).** FTIR spectra for pure OO (—), pure SFO (—) and 50%OO+50%SFO (—)

In this region, for oil samples in initial status, it was registered a maximum at  $1457\text{ cm}^{-1}$  for SFO and at  $1466\text{ cm}^{-1}$  for OO but there was not recorded a linear relationship absorbance versus SFO percent added in pure OO. From this finding results that this frequency is not recommended to be used for evaluation of OO adulteration by SFO addition. This band could be rather used to assess the heating impact on oil quality. Close to this

frequency the double bonds from *trans* isomers don't have absorption while the *cis* isomers from monoenes, dienes and trienes show absorption at the following frequencies:  $1267$ ,  $1265$  and  $1266\text{ cm}^{-1}$ . The band noticed at  $1265\text{ cm}^{-1}$  indicates the existence of unsaturated *cis* isomers without the interference of *trans* isomers.

At a closer inspection of spectral region E it can be noticed two bands near  $1303\text{ cm}^{-1}$  and  $1270\text{ cm}^{-1}$ . First band correspond to the distortion vibration in phase of the methylene group while the second band correspond to the distortion vibration in plane of the  $=\text{CH}$  grouping from the unconjugated double bonds *cis*. We can identify two bands with maximums at  $1378\text{ cm}^{-1}$  and  $1236\text{ cm}^{-1}$ . For oil samples in initial status the absorbance at these frequencies was higher for pure SFO than for pure OO and could be selected for assessing the OO falsification by SFO addition.

In Table 2 are shown the values of absorbance recorded in IR at specific wave numbers for OO, SFO and their mixture in initial status as well as after convective heating. In order to quantify the heating impact on oil quality as well as the degree of OO adulteration by SFO addition we performed the linear regression analysis using the spectral obtained data. Figures 2, 3 and 4 show the correlations between absorbance at  $3011\text{ cm}^{-1}$  (Figure 2),  $3006\text{ cm}^{-1}$  (Figure 3) and  $912\text{ cm}^{-1}$  (Figure 4) versus percent of SFO added in OO. Also, are shown the values of correlation coefficients or Pearson's coefficients (R) obtained as a response to linear regression. These coefficients represent a quantitative measure used for describing of linear relationship strength. By comparing of R values, it was pointed out that there was recorded a significant positive linear correlation for oil samples in initial status. The main disadvantage is that by increasing of heating time the linearity of these curves decreased: after 8 h of heating R values were in the range 0.89-0.95 while after 16 h the value were below 0.7.

The FTIR spectra in region F is characterized by absorption bands specific to the C-C bonds and to the C-O vibration bonds. The carbohydrate chain it is characterized through a series of absorption bands given by vibration of C-C bonds in the ranges  $1100$ - $1000$  and  $900$ - $800\text{ cm}^{-1}$ , while the C-O bond shows two characteristic intense bands at  $1150$ - $1060\text{ cm}^{-1}$  respectively  $970$ - $800\text{ cm}^{-1}$ .

Distortion vibration of carbohydrate chains were detected also close to  $722\text{ cm}^{-1}$  ( $\gamma(\text{C-H})$ ). Absorption in the range  $700$ - $1200\text{ cm}^{-1}$  has shown similar intensity



for all samples in initial status due to the carbohydrate radical from the triglycerides structure of these oils.

For untreated samples as well as for oil samples heated for 8 h it was possible to notice, Table 2, a slight increase of absorbance at 912 and 722  $\text{cm}^{-1}$  with the increasing of the SFO percent added in OO samples. Thus, it is possible to assess the degree of OO falsification by SFO addition using the spectral data recorded at 912  $\text{cm}^{-1}$  on the basis of linear regression absorbance versus percent of SFO added in OO for untreated samples as well as for thermally treated samples for a limited time up to 8 h, Figure 4.

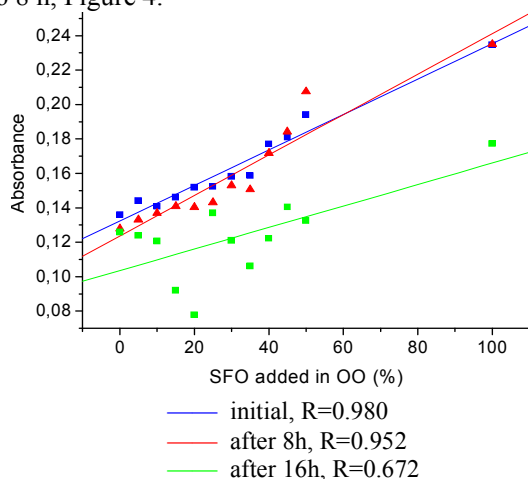


Figure 2. Linear regression analysis absorbance at 3011  $\text{cm}^{-1}$  versus percent of SFO added in OO

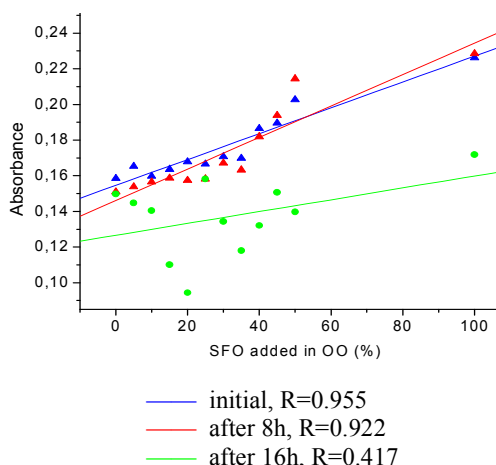


Figure 3. Linear regression analysis absorbance at 3006  $\text{cm}^{-1}$  versus percent of SFO added in OO

Contrary to previous situation, for oil samples subjected to convective heating for 16 h, the spectral data obtained at 912  $\text{cm}^{-1}$  are not useful for

assessing the content of SFO added in OO. From Table 2 it was possible to see that the absorbance values recorded for mixture OO+50%SFO is below the values registered for pure OO and SFO.

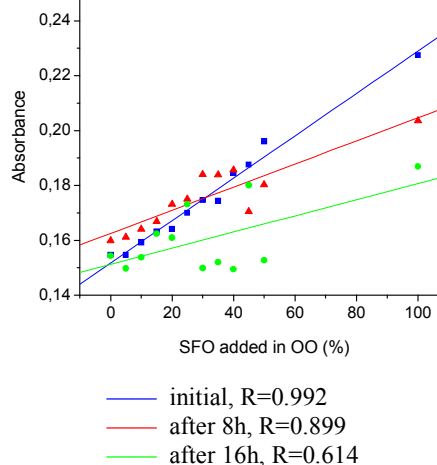


Figure 4. Linear regression analysis absorbance at 912  $\text{cm}^{-1}$  versus percent of SFO added in OO

At the frequency of 722  $\text{cm}^{-1}$  for oil samples in initial status it was noticed a high correlation absorbance versus percent of SFO added in OO. For mixture OO+50% SFO thermally treated for 8h it can be pointed out that the absorbance values is lower the absorbance values recorded for pure OO and SFO. After 16 h of treatment this finding was emphasized being impossible to detect just based of FTIR data the presence of SFO in OO. From the Table 2 it may be noted that in the situation of thermally untreated samples the values of absorbance at 3011  $\text{cm}^{-1}$  has increased linear with the increasing of SFO percent added in pure OO as falsification agent. This finding was in agreement with the results reported by Vlachos *et al.* [17]. After 8 h of heating it was kept the same tendency, proved by the value of R (Figure 2), while after 16 h of heating, due to occurred oxidative degradation it couldn't be further detected the percent of SFO added in OO.

#### 4. Conclusions

Data acquired by carrying out of this study reveal that specific regions of FTIR spectra are very useful for assessing of adulteration and quality of OO. Specific FTIR spectral regions turned out to be very useful for evaluating of OO as well as for the study of the oxidation process during heating at high temperature. Furthermore, spectral changes noticed after heating in the range 3050-2800 and at 1745  $\text{cm}^{-1}$ , help us to monitor the thermally degradation process. The chances in absorbance values at 3011,



3006 and 912  $\text{cm}^{-1}$  show a great importance in order to assess the percentage of SFO added in OO for untreated samples as well as for samples subjected to heating for a limited time up to 8 h. This was possible based on calibration curves absorbance versus percent of SFO added in OO. A significant positive correlation was found especially for oil samples in initial status. In addition, these curves could be useful to estimate the extent of oil alteration as effect of heating. Our data emphasize that FTIR spectroscopy is a simple, fast and low time consuming technique that could be used successfully for evaluate the OO adulteration with cheaper vegetable oil as SFO as well as for assessing the oxidative status of edible oil subjected to heating.

#### Acknowledgments

This study was supported by The Executive Agency for Higher Education, Research, Development & Innovation Funding, Romania (UEFISCDI) through bilateral project ROMANIA-GRECE no. 565/01.06.2012, Program Capacity/module III, Theme: *Rapid spectroscopic methods for assessment of olive oil quality and adulteration*, Project Manager: Assoc. Prof. PhD Mariana-Atena Poiana

#### Compliance with Ethics Requirements

Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human and/or animal subjects (if exists) respect the specific regulations and standards.

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