

Full Length Research Paper

A study on the effect of microwave heating on the properties of edible oils using FTIR spectroscopy

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Fourier transform infrared spectroscopy (FTIR) was used for the structural characterization of vegetable oils (olive, cotton, corn and sunflower oils) following the induced changes in their structure brought about by microwave heating. The vegetable oils samples were microwave heated at 2 powers (450 and 630 W) for different times ranging from 2 - 28 min. The investigation of the FTIR spectra of the treated oils revealed that the microwave heating of oils for different periods of time at 450 and 630 W caused significant changes in the intensities of their absorption bands and produced no shifts in the position of the bands. The results showed that, the microwave heating of oils produced changes in the absorbance ratios $A_{3006}/A_{2924} \text{ cm}^{-1}$ (RI) , $A_{3006}/A_{2854} \text{ cm}^{-1}$ (RII) and $A_{3006}/A_{1746} \text{ cm}^{-1}$ (RIII) which were taken as a measure for the degree of unsaturation. It was found that the changes depended on the applied power, the time of heating and the type of oils. Microwave heating of oils at 630 W for any given time produced an initial decrease in the ratios RI, RII and RIII. This decrease was attributed to the reduction in 18:2 and 18:3 fatty acids content due to the oxidation.

Key words: Edible oils, microwave, FTIR spectroscopy.

INTRODUCTION

Fourier-transform infrared (FTIR) technology has substantial potential as a quantitative quality control tool for food industry (Van de Voort, 1992). The idea of using FTIR spectroscopy to characterize edible fats and oils has been suggested by several authors (Ahmad et al., 1986; Guillen and Cabo, 1998, 1997). Some of them found relationships between the composition of edible oils and lard and the ratios of the absorbance of specific bands of their Fourier transform infrared spectra, and the role of some bands of the fingerprint region (Guillen and

Cabo, 1997).

Microwave heating is a newer cooking method than traditional methods. As a result, the characteristics of microwave heating are not well understood by the public as those inherent in conventional heating. Microwaves have great penetrating power and food products being heated by them have no heat gradients. Foods containing high moisture and fat readily absorb microwaves and are cooked or baked. The application of microwave energy reduces the time for heating when compared to the other methods of heating (Schiffmann, 1992). Many studies have been conducted to investigate the nutritional properties of food treated in microwave oven. Specifically in vegetable oils and fatty foods, there are works about fatty acids isomerization, vitamin loss, their therm-oxidative degradation and stability (Mai et al., 1980; Yoshida et al., 1990; Albi et al., 1997a, b; Thais et al., 1999). The aim of the work was to use FTIR spectroscopy technique for structural characterization of vegetable oils following the induced changes in their composition due to the microwave heating.

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Abbreviation: RI, Absorbances ratios $A_{3006} \text{ cm}^{-1} / A_{2924} \text{ cm}^{-1}$; **RII**, absorbance ratios $A_{3006} \text{ cm}^{-1} / A_{2854} \text{ cm}^{-1}$; **RIII**, absorbance ratios $A_{3006} \text{ cm}^{-1} / A_{1746} \text{ cm}^{-1}$; **RIV**, absorbance ratios $A_{1746} \text{ cm}^{-1} / A_{2924} \text{ cm}^{-1}$; **RV**, absorbance ratios $A_{1746} \text{ cm}^{-1} / A_{1163} \text{ cm}^{-1}$; **Olv**, Olive oil; **Cot**, cotton oil; **Cor**, corn oil; **Sun**, sunflower; **%S**, percentage of saturated fatty acids.

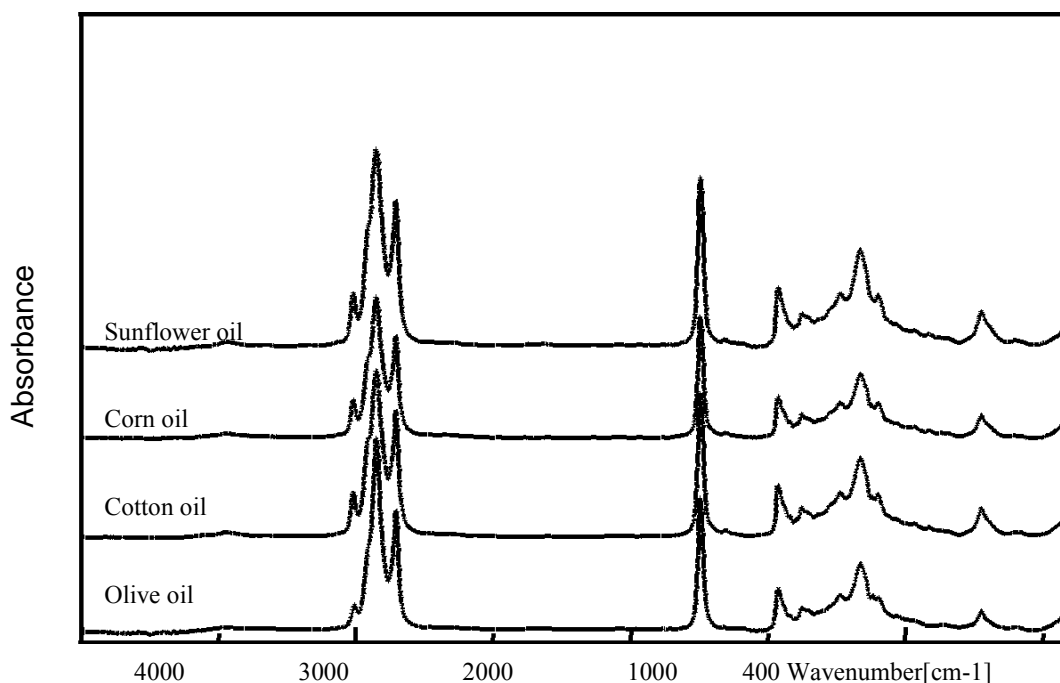


Figure 1. FTIR spectra of edible oil.

MATERIALS AND METHODS

Commercially available olive oil, cotton oil, corn oil and sunflower oil purchased at local market were used in this study.

A film of a small amount of each sample (2 μl) was deposited between two disks of KBr, avoiding the presence of air. For each sample, the spectra were recorded 3 times with 16 scan from 400 - 4000 cm^{-1} for each spectra.

The microwave oven is a Sharp model with 900 W effective power and 2450 MHz frequency. The samples were treated in microwave oven at 2 different powers (450 and 630 W) for different times (2 - 28 min). After microwave heating, the oils samples were cooled immediately at -3°C and stored till measurement.

In the present study, a Fourier transform infrared spectrometer Jasco 430 interfaced to a personal computer operating under windows-based Jasco software was used to record the Fourier transform infrared spectra.

RESULTS AND DISCUSSION

The spectra of untreated oils (olive oil, cotton oil, corn oil, and sunflower oil) are shown in Figure 1. Visual examination of the spectra revealed that there are no appreciable differences between their spectral features apart from slight changes in the absorbances of some bands. All spectra had absorption bands at wavenumber 3471 cm^{-1} (Overtone of -C=O of ester), 3006 cm^{-1} (symmetric vibration of =CH(cis), 2954 cm^{-1} (shoulder)($_{\text{asym}}$ of -C-H of CH_3), 2924 cm^{-1} (asymmetric vibration

of -C-H of CH_2), 2854 cm^{-1} (symmetric vibration of -C-H of CH_2 and CH_3), 1746 cm^{-1} (of -C=O of ester), 1711 cm^{-1} (weak shoulder) (vibration. of -C=O of acid), 1654 cm^{-1} (vibration of cis -C=C-), 1463 cm^{-1} (vibration of -C-H of CH_2, CH_3), 1418 cm^{-1} (vibration of cis =C-H), 1376 cm^{-1} (symetric vibration of -C- H of CH_3), 1238 cm^{-1} and 1163 cm^{-1} (and vibration of -C-O, -CH₂-), 1118 cm^{-1} and 1099 cm^{-1} (vibration of -C -O), 914 (vibration of cis -HC=CH-), and 723 cm^{-1} (vibration of cis - (CH_2)n-, -HC=CH- (Halmiton and Cast 1999; Guillen and Cabo 1997) and (Van de Voort et al., 1995; Safar et al., 1994; Sylverstein et al., 1991 and 1974 ; Ahmad et al., 1986; Guhzler et al., 1975 and Walf and Miwa 1965).

Several authors studied the relation between the absorbances ratios and the degree of unsaturation of fats and oils. Some of them found a close relationship between the degree of unsaturation [the iodine value (IV)] of the edible oils and the absorbance ratio of the band at 3006 cm^{-1} (Arnold and Hartung, 1971). Guillen and Cabo (1998) observed that the IV was related to the ratios A_{3006}/A_{2924} cm^{-1} , A_{3006}/A_{2853} cm^{-1} and A_{3006}/A_{1746} cm^{-1} . The above mentioned absorbances ratios were taken as a measure of the changes in the degree of unsaturation of the oils.

Table 1 gives the absorbance ratios RI, RII, RIII and the iodine values (as reported from literatures) (Hui, 1979) of olive, cotton, corn and sunflower oils. As shown in Table 1, the olive oil had the lowest values of the absorbance ratios, while sunflower oil had the highest

Table 1. The absorbance ratios RI, RII and RIII together with the iodine value (IV) for the experimental oils as reported in the literature (Hui, 1979).

Oil	IV range	RI	RII	RIII
Olive	84 - 90	0.1291 ± 0.011	0.1951 ± 0.008	0.1810 ± 0.009
Cotton	90 - 117	0.2031 ± 0.018	0.2998 ± 0.013	0.2449 ± 0.016
Corn	103 - 128	0.2017 ± 0.020	0.3135 ± 0.015	0.2499 ± 0.017
sunflower	125 - 136	0.2989 ± 0.061	0.4039 ± 0.053	0.3432 ± 0.057

one. This result could be attributed to the fact that olive oil has the lowest value of degree of unsaturation (that is to say iodine value IV) among the other oils under investigation. The iodine values of the oils reported in literature (Table 1) confirms this result. The trend of the values of RII is well consistent with the IV values reported in the literatures.

Careful examination of the spectra of the microwave heated samples revealed that the microwave heated oils under analysis for any given time and at either 450 or 630 W caused slight changes in the intensities of the most evident absorption bands.

The relationship between the absorbance ratios RI, RII and RIII of the oils used before and after microwave heating at 450 and 630 W and the time of treatment had the same trend. Figure 2 illustrates the relationship between RII and time of treatment as. From Figure 2a, it can be seen that, heating of the olive oil for 2 - 16 min at 450 W increased the absorbance ratios, whereas, heating for several times above 16 min decreased them. The maximum value of the ratios appeared at 16 min. The microwave heating of the cotton oil at 450 W either increased or decreased the absorbance ratios depending on the time of heating. The maximum value of the ratios appeared at 16 min and the minimum value at 28 min. Also, the absorbance ratios of the microwave heated corn oil at 450 W suggested maximum and minimum values according to the time of heating. The maximum appeared at 12 min and the minimum at 2 min. The microwave heating of the sunflower oil at 450 W resulted in either remarkable decrease or increase in the absorbance ratios depending on the time of treatment. The smallest value of the ratios was observed at 2 min and the greatest value at 16 min of the exposure time. The microwave treatment of all types of the experimental oils for different times at 630 W (Figure 2b) produced a considerable decrease in the absorbance ratios (except for the corn oil sample treated for 28 min which had a high increase in the absorbances ratios).

From the above mentioned considerations, one can conclude that the increase in the absorbance ratios which reached its maximum at 16 min for olive, cotton and sunflower and at 12 min for corn oil provided a strong evidence for the increase in the degree of unsaturation due to the increase in cis double bonds. This increase may be attributed to the formation of free radicals, as a result of

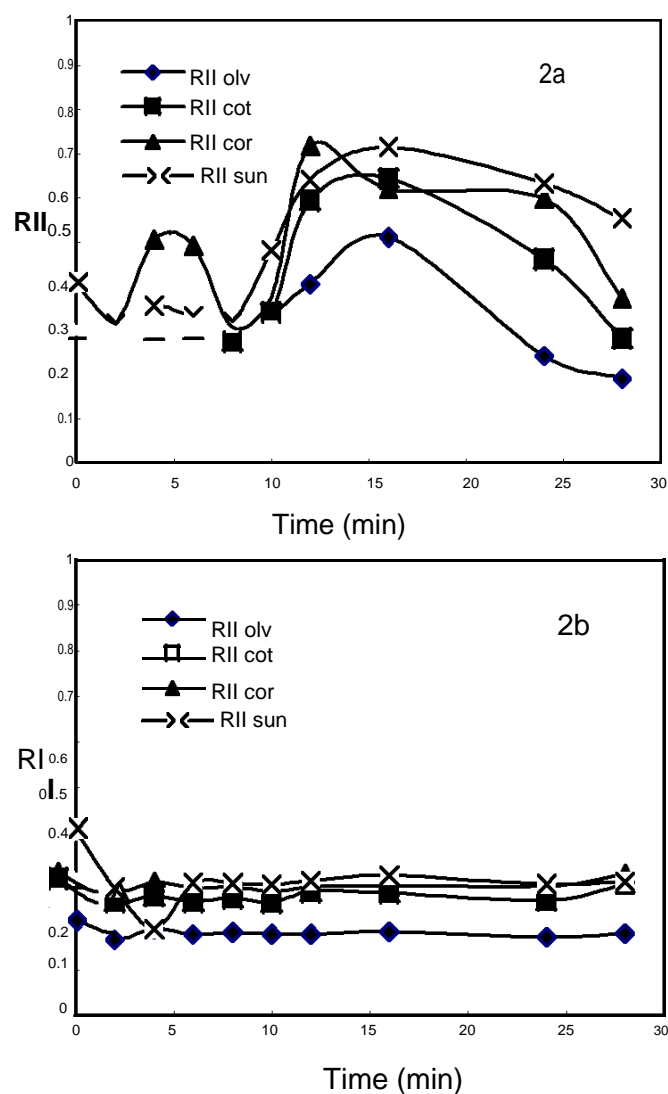


Figure 2. The variation of the absorbance ratio RII of unheated and microwave heated olive (olv), cotton (cot), corn (cor) and sunflower (sun) oils treated at (a) 450 W and (b) 630 W for different times.

microwave heating that initiate primary oxidation reaction of unsaturated fatty acids. This reaction resulted in primary oxidation products that contained double bonds in cis form and conjugated double bonds as what

happened in the case of the autoxidation of oleic and linoleic acid (Belitz and Grosch, 1999). This result interpretation agrees with that reported in literatures (Yoshida et al., 1990; Thais et al., 1999; Farag et al., 1992). Also, the decrease observed after 16 min for olive, cotton and sunflower and at 12 min for corn oil may be due to the fact that, the rate of the formation of the primary oxidation products was lower than their rate of degradation after 16 min for olive, cotton and sunflower and 12 min for corn oils. The degradation of primary oxidation products produces secondary oxidation products (aldehydes, ketones, acids, and esters) and this resulted in a decrease in the degree of unsaturation due to the decrease of cis double bonds of the compounds. This result is confirmed by that obtained by Moreno et al. (1999). It can be seen that the initial decrease in the absorbance ratios that occurred at 2 min and from 2 - 8 min of time exposure for corn and sunflower, respectively may be due to the decrease in the degree of unsaturation which may be attributed to the reduction in the 18:2 and 18:3 fatty acids (unsaturated fatty acids in general) due to the oxidation. This result agrees well with that found by Yoshida et al. (1990), Paquette et al. (1985) and Van de Voort et al. (1994). The decrease in the degree of unsaturation (primary oxidation products) at 8 min heating of corn oil may be attributed to either the free radicals (that contains cis double bonds) that are scavenged by its antioxidants such as tocopherols or the break down of the primary oxidation products to form secondary oxidation compounds. Niki et al. (1982) reported that the effectiveness of tocopherols as lipid antioxidants has been attributed mainly to their ability to break chain reactions by reacting with fatty acid peroxy radicals. The microwave treatment of oils at 630 mW caused a decrease in the degree of unsaturation. This decrease is an indication of the decrease in the 18:2 and 18:3 fatty acids (unsaturated fatty acids) due to the oxidation. This result agrees well with the results of Yoshida et al. (1990) and Van de Voort et al. (1994).

The relation between absorbance ratio $A_{1746}/A_{2924} \text{ cm}^{-1}$ (RIV) of the untreated and microwave treated experimental oils at 450 (3a) and 630 W (3b) and the time of treatment indicated that, the microwave treatment of the olive oil for all times at 450 W produced a considerable increase in the value of this ratio. The maximum value of this increase appears at 12 min of exposure while the minimum one was observed at 4 min of the exposure time. For the other oils, the microwave exposure at 450 W caused a decrease and increase in the absorbance ratio. The extent of the decrease and increase varied according to the type of the oil and the time of treatment. For the cotton oil, this decrease was very slight for the samples treated for 2, 4 and 28 min, respectively. The only very slight decrease for the microwave heated corn oil at 450 W appeared at 2 min of exposure time, while a remarkable increase was noticed for the other times of the experiment. This increase

reached its highest value for the sample treated at 16 min while the lowest increase occurred for the sample heated for 8 min. On the other hand, the microwave heating of sunflower at 450 W produced a slight decrease in the absorbance ratio for sample treated for 2 and 6 min. A slight increase in the absorbance ratio was seen for the samples treated for other exposure time. The maximum value of this increase was present at 16 min of the treatment while the minimum value was observed at 28 min of exposure. The microwave heating for the experimental oils for different times at 630 W produced an initial decrease in the absorbance ratios after 2 min for all oils samples; this decrease remained more or less constant until 28 min for olive and sunflower and for 24 min for cotton and corn oils. Heating after 24 min caused a marked increase in the above mentioned absorbance ratios of the cotton and corn oils. The extent of the decrease and increase varied according to the type of oil and the time of treatment. The highest value of this decrease for all the oils used appears at 4 min of exposure time while the lowest value appeared at 28 min for olive and sunflower oil.

The obtained results indicate that the microwave heating of oil samples at 450 W resulted in an increase and decrease in the carbonyl groups (as shown from the changes in the absorbances ratio). This increase may be due to the formation of secondary oxidation products such as aldehydes, alcohol, ketones, acids and esters (Frankel, 1982), while the decrease may be attributed to the evaporation of some compounds of the secondary oxidation compounds and the hydrolysis of triglyceride as a result of microwave heating. On the other hand, the decrease in the carbonyl groups of oils microwave heated at 630 W (Figure 3b) may be attributed to the dissociation of its original carbonyl group esters due to the hydrolysis of triglycerides as a result of the microwave exposure. This result is confirmed by the result of Yoshida et al. (1992) who reported that microwave heating caused a random hydrolysis of triglycerides. The less decrease obtained for samples of all oils microwave heated at 630 W for time longer than 4 min may be due to the formation of secondary oxidation products such as aldehydes, ketones, alcohols, acids and esters. Also, the high increase that occurred in the samples of cotton and corn oils treated for 28 min may be attributed to the high increase in the formation of the secondary oxidation products. It can be seen that the changes in the absorbance ratio RIV of the treated oils may be due to the oxidative and hydrolysis processes as a result of microwave heating.

Figure 4 illustrates the relation between the values of the absorbance ratio $A_{1746}/A_{1163} \text{ cm}^{-1}$ (RV) of the untreated and microwave treated oils under investigation at 450 and 630 W and the time of treatment. From Figure 4a, it can be seen that, the microwave treatment of the olive oil at 450 W for any time of exposure, resulted in a remarkable decrease in the absorbance ratios except for

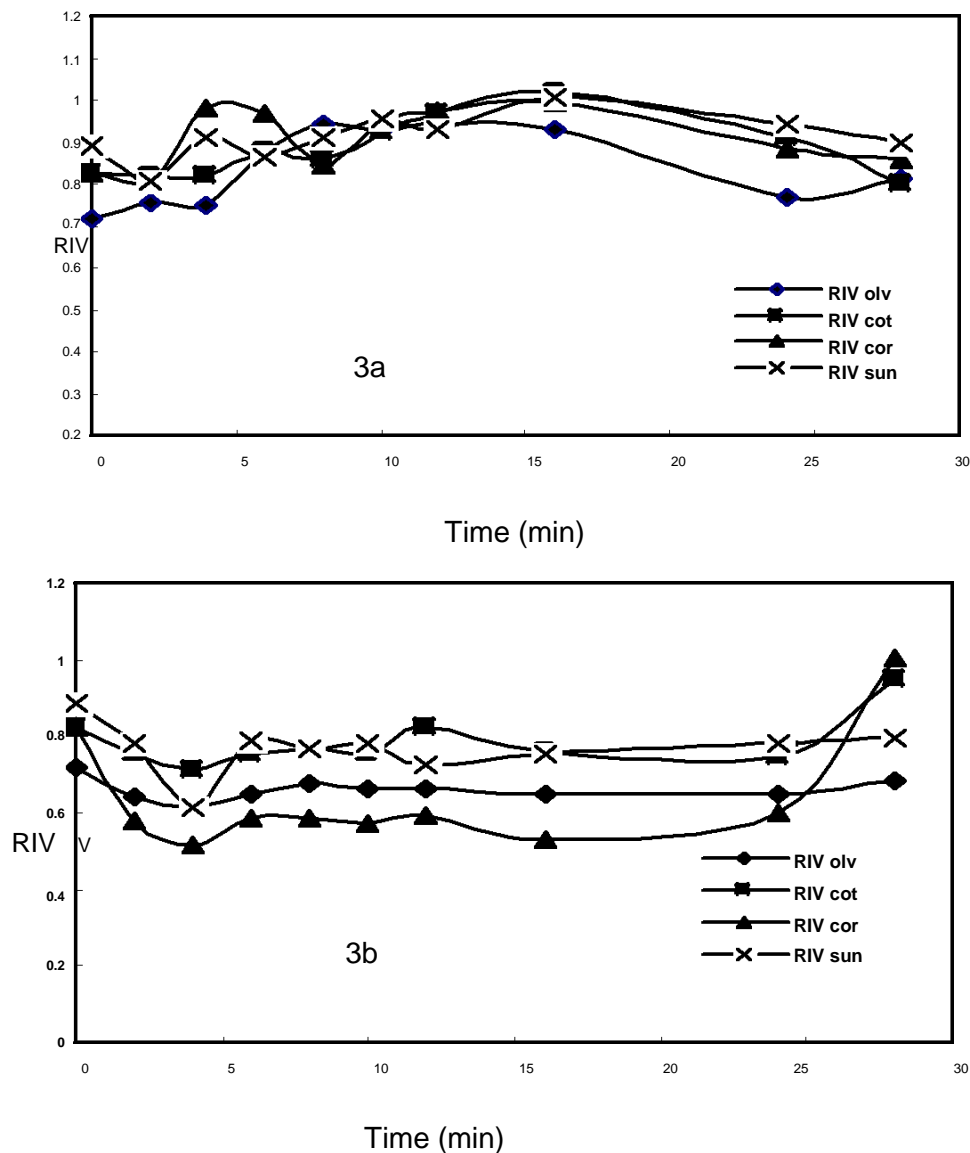


Figure 3. The variation of the absorbance ratios $A_{1746}/A_{2924\text{cm}^{-1}}$ (RIV) for unheated and microwave heated oils with exposure time at (a) 450 W and (b) 630 W.

the sample treated at 4 min in which very slight increase was observed. This decrease reached its maximum value after 16 min of treatment while its minimum value was observed after 28 min of treatment. Heating of the cotton oil by microwave at 450 W decreased (except for the samples heated at 8 and 28 min) the absorbance ratio. The highest value of decrease was noticed at 16 min of treatment, whereas, its lowest value was observed at 4 min. Microwave heating of the corn oil at 450 W for 2 and 8 min produced a considerable increase in the absorbance ratio. For the other exposure times a remarkable decrease appeared; its maximum value was observed at 12 min while its minimum one was seen at 28 min of exposure time. On the other side, the

microwave heating of the sunflower oil samples treated up to 8 min at 450 W resulted in a remarkable increase in the absorbance ratio. The highest value of this increase was found at 2 min, whereas, the lowest one was observed at 4 min of treatment. Heating for time longer than 8 min produced a considerable decrease in the value of the ratio, its maximum value was observed at 16 min and the minimum value existed at 28 min. The absorbance ratio RV assumes its highest value of decrease after heating cotton oil for 16 min at 450 W. For any given time, the microwave heating of the oil under analysis at 630 W (Figure 4b), resulted in a marked increase in the value of the absorbance ratio RV.

The obtained results show that in the case of the

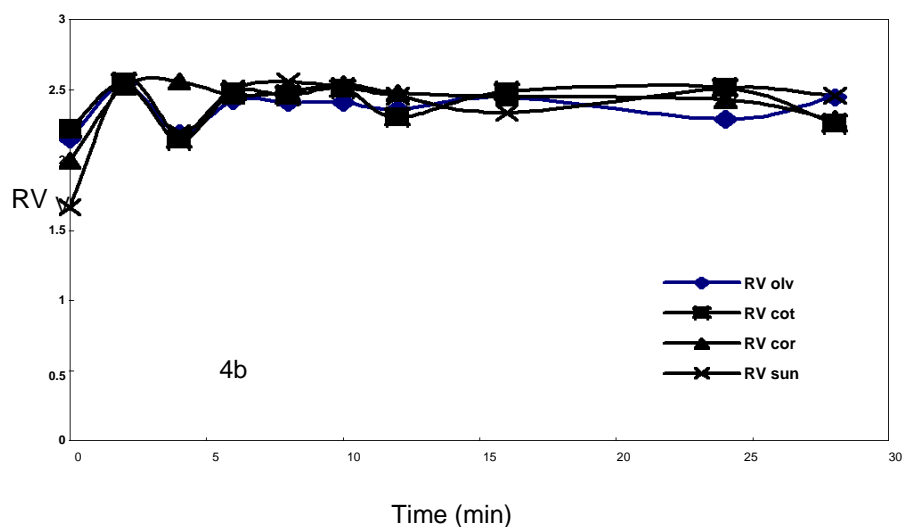
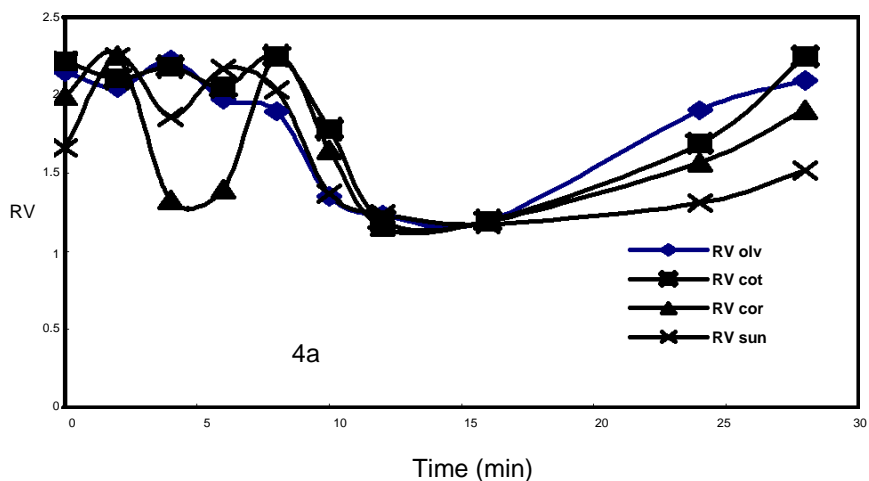


Figure 4. The variation of the absorbance ratio $A_{1746}/A_{1163\text{cm}^{-1}}$ (RV) for unheated and microwave heated oils with exposure time at (a) 450 W and (b) 630 W.

microwave heating of oils at 450 W, the maximum decrease in the absorbance ratio that appeared at 16 min for olive, cotton and sunflower at 12 min for corn oil was attributed to the decrease in %S. This decrease may be due to the increase in the acids containing double bonds, or conjugated double bonds, formed during the oxidative primary reactions of unsaturated fatty acids. The increase in the absorbance ratio that occurred at 2 and 8 min of treatment of corn oil and from 2 - 8 min of heating time for sunflower may be due to the break down of the formed acids either by more oxidation or their degradation. Also, the initial increase in the absorbance ratio of all the oils heated for different periods of time at 630 W indicated that the increase in %S was as a result of the oxidation process. The last results are in agreement with that obtained from literatures (Yoshida et al. 1990, 1995, 1997). The result of the above mentioned ratio support the results obtained from the ratios representing the

degree of unsaturation which are described before.

Conclusion

It can be concluded that FTIR spectroscopy can be used for differentiation between the different oils. The microwave heating of oils caused oxidation and hydrolysis of the oils and the processes occurred more rapidly in the case of the oils treated at 630 W than those treated at 450 W. The FTIR spectroscopy is a good tool for following these changes in oils.

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