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Rheological Properties and FTIR Spectra of Olive, Chia and Garden Cress Oils Exposed to High Temperatures for Different Times

Ghada H. Shahda[1,2], Sayed M. Mokhtar[1], Ramadan A. Habiba[1] and Khaled M. Youssef[1*]

[1] Food Technology Department, Faculty of Agriculture, Suez Canal University, 41522 Ismailia, Egypt
[2] Home Economic Department, Faculty of Specific Education, Port Said University, Port Said, Egypt

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Abstract: Rheological properties, peroxide value, and FTIR spectra of olive, chia and garden cress oils exposed to high temperatures (150, 175, and 200 ºC) for different times (30, 60, and 120 minutes) were assayed. The shear stress versus shear rate data (at 25 ºC) was fitted to Newtonian, Bingham, and power law rheological mathematical models. Fresh tested oils showed Newtonian flow behavior with correlation coefficients higher than 0.99 and slight non-Newtonian behavior after heat treatment at different temperatures and times. The peroxide values of the tested oils were significantly increased with increasing the temperature and time, excepting the garden cress oil treated at 200 °C. The viscosity is more related to the peroxide value of olive and chia oils (R^2= 0.8844 and 0.9768, respectively) than that of garden cress oil (R^2= 0.7325). The FTIR spectra showed little decrease in absorption at 2924 and 2855 cm^{-1} in olive and garden cress oils, and at 1744 cm^{-1} in chia oil after thermal treatment. The vibrations appear at 3200 – 3500 cm^{-1} in chia and garden cress oils indicating the presence of peroxides produced during thermal treatment, while it disappeared in olive oil. A peak at 1672 cm^{-1} was noticed in olive oil indicated the presence of free fatty acids. A slight difference was observed at 962 cm^{-1} between spectra for chia and garden cress oils, indicating the formation of *trans* fatty acids. In conclusion, the rheological properties and FTIR spectra can be used as good tools to estimate the quality of heated oils.

Keywords: chia oil, garden cress oil, olive oil, rheological properties, peroxide value, FTIR spectra

1. Introduction

Plant oils are vital for providing essential fatty acids, fat-soluble vitamins (K, A, D, and E), energy, and enhancing metabolism (Dorni, *et al.* 2018 and Zhao, *et al.* 2021). A healthy diet should contain plant oils that are low in saturated fatty acids and high in monounsaturated and polyunsaturated fatty acids (Konuskan, 2019). Excess consumption of fat-containing foods high in saturated and trans fatty acids has been linked to a higher risk of non-communicable diseases such as coronary heart disease, hypercholesterolemia, metabolic syndrome, stroke, and obesity (Hooper, 2020). Oppositely, polyunsaturated fatty acids play an important role in the prevention of chronic diseases such as hypertension, coronary artery disease, and cancer (Poudyal, 2012).

Oils are commonly used for food processing and domestic cooking. They serve a dual purpose

* Corresponding author: khaled_youssef@agr.suez.edu.eg

in the production of fried foods; they assist as a heat transmission medium between fryer and food, and they play an important role in the flavor and texture of foods (Tseng, *et al.* 1996 and Aniołowska & Kita 2016). Previous studies suggest that the quality of both oils and fried foods is directly influenced by the type of oil, temperature, and time as thermal exposure alteration chemical and physical properties of plant oils (Ma, *et al.* 2021 and Liu, *et al.* 2021).

Lipid oxidation is a major source of quality degradation, which causes nutritional loss, reduced shelf-life, formation of off-flavors and increased rancidity (Galano, 2015). Furthermore, secondary oxidation products like aldehydes and ketones can build up in highly oxidized oils, which may have harmful and carcinogenic properties (Min & Boff, 2002 and Vieira, *et al.* 2017). Frying is a popular and long-established cooking technique used to prepare meals all over the world. Improper thermal treatment can have undesirable effects such as generation of hazardous chemicals such as furans and acrylamides, and oil oxidation which raises peroxide levels (Brühl, 2014). It is expected that the presence of higher unsaturation in frying medium is closely associated with the oxidative deterioration of plant oils. To improve the oxidative stability of plant oil when exposed to higher temperatures, low unsaturation is recommended. As higher level of oleic acid in frying medium is demonstrated to give higher stability during frying process (Bou, *et.al.* 2012 and Romano, *et al.* 2021) such as olive oil. Consequently, it is essential to use the right oil for cooking (Makni, *et al.* 2015).

The indicators of oil quality in frying oil include viscosity and others such as color measurements, peroxide value, density, etc. (Jacobson, 1991). Temperature has a significant impact on an oil's viscosity. When heated, the oil may get more viscous. This appears to be caused by isomerization, hydrolysis, oxidation, and the polymerization process (Lin, *et al.* 1998). In plant oils, viscosity increases with triglyceride fatty acid chain length and reduces with unsaturation. Thus, the dimension and orientation of molecules determine their viscosity (Hassanien & Sharoba 2014). Kim *et al*. (2010) reported that viscosity has a positive correlation with monounsaturated fatty acids and a negative correlation with polyunsaturated fatty acids. Plant oils are reported to be shear thinning at low shear rates and becoming Newtonian at higher shear rates above 200 s^{-1} (Kim, *et al.* 2010).

Many oils have been tested for their thermal stability and rheological characteristics such as sesame, mustard, sunflower and cottonseed oil (Hassanien & Sharoba 2014, Stanciu, 2014 and Valantina, *et al.* 2015). There is little information about the thermal stability and rheological properties of promising plant oils, such as chia and garden cress seed oils. Thus, the objectives of the present study were to evaluate the rheological properties, peroxide value, and FTIR spectra of chia and garden cress oils and olive oil as a reference oil and investigate the correlation between viscosity and peroxide value of these oils during exposure to high temperatures (150, 175, and 200 ºC) for different times (30, 60, and 120 minutes).

2. Materials and Methods

2.1. Raw materials

Garden cress (*Lepidium sativum*) seeds, chia (*Salvia hispanica* L.) seeds, and olive (*Olea europaea* L.) fruits were all purchased from a local marketplace (Fayoum City, Egypt). The different seeds and fruits were cleaned from dust and plant debris.

2.2. Oil extraction by mechanical pressing

Oils were extracted from the raw materials by screw pressing. The extraction by mechanical pressing was carried out with a hydraulic cold pressing machine (Kern Kraft, Germany) at room temperature (28±2 ºC), and at pressure of 10 MPa for 10 min according to the method of Uquiche, *et al.* (2008). The extracted oils were filtered, and then centrifuged at 3500 rpm for 20 min in order to separate the components that were settled. The obtained oils were stored at -18 °C till further analyses.

2.3. Heating treatment

The olive, garden cress and chia oils samples were heated at 150 °C, 175 °C, and 200 °C in an oven (WT-binder, Type F115, Germany) for 30, 60, and 120 minutes. The experiment was performed in brown glass bottles without covering. After cooling, the bottles were closed and the samples were stored in dark at 4 ºC till further analyses.

2.4. Determination of peroxide value

The peroxide value (PV) of examined oils was determined and calculated by method reported by Kharbach, *et al.* (2021). One gram of sample was added in a 250 ml Erlenmeyer flask. The sample was mixed thoroughly with 30 ml of glacial acetic acid: chloroform (3:2, v/v), and 1 ml of saturated potassium iodide solution. The mixture was kept for 2 min in dark, after which 30 ml of distilled water and 1 ml gelatinized starch solution were added, and titrated with $Na_2S_2O_3$ 0.01 *N*. The peroxide value was calculated with the following equation:

$$PV = V_{Na2S2O3} \text{ x } N_{Na2S2O3} \text{ x } 1000 / m \qquad (1)$$

where V represents the volume of $Na_2S_2O_3$ used for titration, *N* normality of the solution used for titration, and *m* the weight of the sample in grams.

2.5. Fourier transfer infrared (FTIR) spectral

The FTIR spectra of chia, garden cress and olive oil were determined by FTIR spectrometer (Bruker Optics, Germany) in the range of 4000 - 500 cm^{-1} before and after thermal treatment. Transmittance mode was selected for measurement. The empty cell was used as the blank. The cell was cleaned with tissue paper soaked in chloroform after measurement and before measuring a new sample. The measured spectra were further manipulated using IR solution software provided with the instrument and OriginPro software (OriginLab Corporation, Northampton, UK (Hasan & Khan 2020 and Mitrea, *et al*. 2020).

2.6. Rheological properties of chia, garden cress and olive oils

The rheological properties of different oils were evaluated before and after thermal treatment by the Brookfield Digital Rheometer model DV-III+. The Brookfield small sample adapter (8 ml sample) and $Sc_{4\text{-}21}$ spindle were used. Temperature was maintained using a thermostatically controlled water bath at 25 ºC. The shear rate was varied from the lowest value (0.93 s^{-1}) to the highest value (232.5 s^{-1}), where 25 rotational speeds were used. All data were taken after 10 second between every rotational speed in each sample.

The shear stress versus shear rate data was fitted to Newton's model (Equation 2) by linear regression, Bingham plastic (Equation 3), and Power Law (Equation 4) mathematical models [19].

$$\boldsymbol{\tau = \eta\gamma} \qquad (2)$$

where τ is the shear stress (mPa), η is dynamic viscosity (mPa.s), and γ is the shear rate (s^{-1}).

$$\boldsymbol{\tau = \tau_{oB} + \eta_B\gamma} \qquad (3)$$

$$\boldsymbol{\tau = K\gamma^n} \qquad (4)$$

where τ_o is the yield stress (mPa), shear stress at zero shear rate, η_B is plastic viscosity (mPa.s), K is consistency multiplier (mPa.s^n), and n is flow behavior index.

2.7. Statistical analyses

One-way ANOVA is operated to evaluate the data, which are shown as mean of three replicates. Duncan’s test was employed to determine the difference between treatment means. All statistical analyses were conducted by the statistical program SPSS 11.0 (SPSS Ltd., UK). Means separation at $p \leq 0.05$ was deemed to be statistically significant.

3. Results and Discussion

3.1. Changes of peroxide value during heating treatment of the olive, chia, and garden cress oils

Peroxides content is given by the degradation of oils past oxidation and indicates the milli-equivalents of oxygen that reacts with one kilogram of oil and release iodine from the sodium thiosulfate solution (Varona, *et al.* 2021 and Mitrea, *et al.* 2022). Peroxide value with acid value and density and some other characteristics values can used together to make a clear picture about the freshness of oil samples, where changes these values from the standard values are related with unsuitable storage or processing conditions (Alimentarius, 2021 and Mitrea, *et al.* 2022). Oils that are commercially accessible should have a maxim level up to 10 meq O_2/ kg oil of peroxide value [30]. As it can be noticed from Table (1), there are differences among the peroxide values of the tested samples ($p \leq 0.05$). From the obtained results, the oil samples exposed to thermal treatment for 30 min exceeded the standard limits for peroxide value, excepting the garden cress oil treated at 150 and 175 °C where it had the lowest values (7.96 and 8.03 meq O_2/ kg oil) among the analyzed samples.

Also, the peroxide values of the tested oils were significantly increased with increasing the temperature of the thermal process and the time, excepting the garden cress oil treated at 200 °C for different times. The highest peroxide values were recorded for chia oil. This may be due to oxidation of PUFAs found at high concentration in this oil (more than 80%) as reported in many researches. The decreasing of peroxide value for garden cress oil heated at 200 °C for 60 and 120 min may be due to the degradation of the resultant peroxides to aldehydes and ketones. Ghobadi *et al*. (2018) analyzed more than 42 samples of fried oils (unspecified oil sample, and fried temperature was 180 °C) collected from restaurants and fast-foods units. Their results exceeded the standards limits as they obtained a mean peroxide value of 41.5 meq O_2/ kg oil. Mitrea *et al*. (2022) heated sunflower, rapeseed, corn, palm, and coconut oils at 180 °C for 30 min and found that the peroxide value exceeded the standards limits for all examined oils ranged from 12.07 meq O_2/ kg oil for coconut oil to 50.71 meq O_2/ kg oil for palm oil sample. Manzocco, *et al*. (2020) found that the peroxide value of sunflower oil maintained for 2 days at temperature higher than 40 °C exceeds the standard limits (25.22 meq O_2/ kg oil) and develop a rancid odor. Also, Giuffré, *et al*. (2018) studied the effect of heating and time exposure to raised temperatures (180 and 220 °C for 30, 60, and 120 min) on peroxide value for four vegetable oils (extra virgin olive oil, pomace olive oil, soybean oil, and palm oil). They concluded that as much as the time exposure to heating rises, the more increases the peroxide value. The peroxide value for extra virgin oil increased from 8.1 to 20.9 meq O_2/ kg oil after heated 120 min at 220 °C. Pomace oil and soybean oil presented lower peroxide values after heated for 120 min at 220 °C (11.8 and 9.5 meq O_2/ kg oil, respectively). Palm oil showed the lowest increase in peroxide value (6.4 meq O_2/ kg oil) after processed for 120 min at 220 °C.

Table 1. Changes of peroxide value (meq O_2/ kg oil) of olive, chia, and garden cress oils during heat treatment at different temperatures and times

Oil sample	Heating temperature (°C)	Heating time (min)	Peroxide value
Olive	150	30	14.67^{j}
		60	16.00^{i}
		120	20.37^{g}
	175	30	15.14^{ij}
		60	17.80^{h}
		120	22.37^{e}
	200	30	14.50^{j}
		60	17.98^{h}
		120	21.71^{ef}
Chia	150	30	17.17^{h}
		60	20.98^{fg}
		120	35.83^{a}
	175	30	20.69^{fg}
		60	22.50^{e}
		120	32.26^{b}
	200	30	20.96^{fg}
		60	21.24^{fg}
		120	33.05^{b}
Garden cress	150	30	7.96^{m}
		60	10.35^{l}
		120	29.38^{c}
	175	30	8.03^{m}
		60	15.05^{ij}
		120	24.08^{d}
	200	30	11.11^{kl}
		60	11.34^{kl}
		120	11.53^{k}

Values are means of n=3

Means with different character are significantly different at $p \leq 0.05$

3.2. Rheological characteristics of studied oils during heating treatment

The changes in viscosity of heat treated oil are the signs of oil deterioration. The oil may thicken and become more viscous as it is heated. This seems to be due to the process of polymerization and to oxidation, hydrolysis and isomerization (Lin, *et al*.1998). Viscosity measurements can provide an overall estimate of heated oil quality (Jacobson, 1991).

Olive, chia, and garden cress oils samples showed great changes in viscosity and rheological parameters calculated by Newtonian, Bingham, and power law models after heated at different temperatures (150, 175, and 200 °C) and times (30, 60, and 120 min) as shown in Table (2). As the oxidation accelerated by heat proceeded, the values of viscosity progressively increased and these results are in agreement with (Lin, *et al.* 1998 and Chatzilazarou, *et al.* 2006). Garden cress oil showed higher increase in viscosity followed by chia oil then olive oil. These results clearly indicate the deteriorative effect of oxidation and polymerization of heated oils compared to fresh oils. Viscosity of oil is strongly affected by its degradation products, increasing as a result of formation of dimers, trimers, polymers, epoxides, alcohols and hydrocarbons (Stevenson, *et al.* 1984). The increase in viscosity of oils was due to polymerization which resulted from the formation of high molecular weight compounds (carbonto-carbon and/or carbon-to-oxygen-to-carbon bridges) between fatty acids (Chatzilazarou, *et al.* 2006 and Maskan, 2003). The viscosity of the oil changes considerably with heating time and this change must be taken into consideration when designing frying operations to control the product quality.

Table 2. Rheological parameters of olive, chia, and garden cress oils during heating process at different times using the Newtonian, Bingham and power law models

Oil sample	Heating temperature (°C)	Heating time (min)	Newtonian model		Bingham model			Power law model		
			η	R^2	σ_{0B}	η_B	R^2	K	N	R^2
Olive	Control		64.2	0.9995	0.12	63.3	0.9999	118.2	0.86	0.9906
	150	30	63.8	0.9939	0.37	61.2	0.9980	121.7	0.88	0.9693
		60	67.5	0.9995	0.12	66.7	0.9998	123.2	0.86	0.9817
		120	72.2	0.9999	0.07	71.7	1.0000	98.6	0.93	0.9979
	175	30	66.9	0.9998	0.07	66.4	1.0000	91.6	0.93	0.9982
		60	67.6	0.9999	0.05	67.3	0.9999	79.1	0.97	0.9951
		120	73.1	0.9997	0.10	72.4	0.9999	98.9	0.94	0.9889
	200	30	66.8	0.9997	0.09	66.2	0.9936	81.6	0.96	0.9936
		60	68.0	0.9998	0.06	67.5	0.9999	78.9	0.97	0.9947
		120	72.3	0.9999	0.06	71.9	1.0000	78.8	0.99	0.9964
Chia	Control		37.3	0.9995	0.07	36.8	0.9999	63.3	0.89	0.9958
	150	30	38.6	0.9993	0.08	38.1	0.9998	64.7	0.89	0.9937
		60	40.9	0.9998	0.04	40.6	0.9999	84.4	0.89	0.9919
		120	52.3	0.9995	0.10	51.6	0.9999	89.3	0.88	0.9924
	175	30	41.3	0.9959	0.21	39.9	0.9988	131.1	0.75	0.9670
		60	42.6	0.9991	0.09	42.0	0.9996	74.9	0.88	0.9890
		120	52.2	0.9998	0.05	51.8	0.9999	66.1	0.95	0.9962
	200	30	40.3	0.9999	0.02	40.1	0.9999	49.2	0.96	0.9986
		60	41.4	0.9995	0.01	41.3	0.9995	42.8	0.99	0.9704
		120	50.6	0.9999	0.01	50.5	0.9999	52.7	0.96	0.9917
Garden crcress	Control		53.2	0.9996	0.09	52.6	0.9999	80.8	0.91	0.9924
	150	30	54.4	0.9993	0.11	53.7	0.9998	81.0	0.92	0.9918
		60	57.5	0.9996	0.09	56.9	0.9998	85.4	0.91	0.9823
		120	77.6	0.9999	0.06	77.3	1.0000	82.1	0.99	0.9954
	175	30	55.4	0.9997	0.05	55.0	0.9998	69.0	0.95	0.9924
		60	57.6	0.9988	0.11	56.8	0.9992	87.3	0.91	0.9785
		120	66.9	0.9999	0.03	66.7	1.0000	73.2	0.98	0.9973
	200	30	55.7	0.9993	0.10	55.0	0.9997	79.5	0.93	0.9927
		60	60.6	0.9971	0.38	58.6	0.9996	119.0	0.87	0.9809
		120	67.7	0.9998	0.07	67.6	0.9999	71.2	0.99	0.9931

η= viscosity (mPa s), σ_{0B}= yield stress (N/ m^2), η_B= plastic viscosity (mPa s), K= consistency coefficient (mPa s^n), n= flow behavior index, R^2= determination coefficient, Values are means of n=3

The shear stress versus shear rate data was fitted to Newtonian, Bingham and power law rheological models. The flow behavior of fresh and heated oil samples was measured at 25 °C. Fresh oils showed Newtonian behavior with correlation coefficients greater than 0.99 and slight non-Newtonian behavior after heat treatment at different temperatures and times. These results are in agreement with those of Santos, *et al*. (2005) and Hassanien and Sharoba (2014) who found that the flow behavior of some edible vegetable behaved as non-Newtonian fluids after frying for different times. The fresh oils are Newtonian liquids having high viscosity due to their long chain structure. The results presented in Table (7) indicated that the studied oils still showed Newtonian flow behavior after heating with little Bingham behavior (R^2 higher than 0.99) and not fitted to power law model indicated no pseudoplastic behavior.

Viscosity of oils increased during heat treatment and was influenced by heating time and changes in viscosity indicated significant structural change. The tendency of viscosity to increase during heating of the oil has been found to correlate with formation of polymers (Gloria and Aguilera 1998). The increasing in viscosity indicates that polymers, which are higher molecular weight fraction of the degradation products, are increased by increasing heating time. Also, increasing in viscosity may be due to the effect of free fatty acids produced by hydrolysis reactions and other small molecular weight decomposition products during heating (Santos, *et al*., 2005). The differences in viscosity after thermal exposure appear because of changes in the chemical profile, especially when the content of unsaturated fatty acids is diminished (Ghazani, *et al.* 2015 and Mitrea, *et al.* 2022).

The rheological behavior of oils samples is of special importance when they are used to study the relation between the chemical properties and the flow behavior of oils. These properties are sometimes measured as an indicator of product quality (e.g., indication of type of oil or change in molecular size). The correlation between peroxide value presented in Table (1) and the Newtonian viscosity recorded in Table (2) is shown in Figure (1).

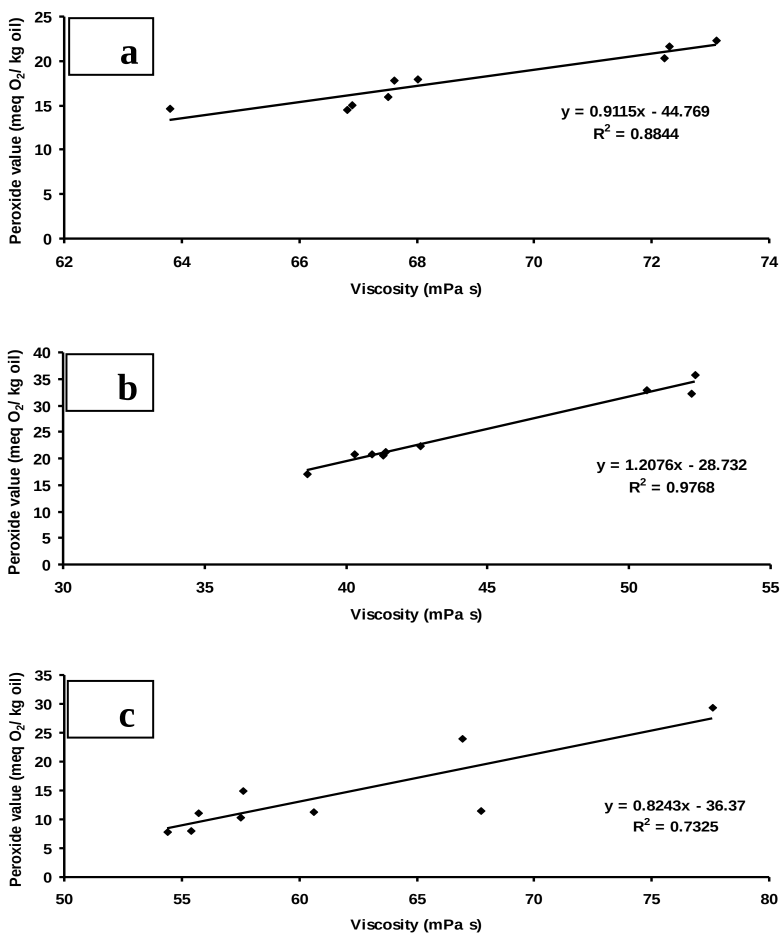

Figure 1. Correlation between viscosity and peroxide value a) olive oil, b) chia oil, and c) garden cress oil

The results indicated that viscosity is more related to the peroxide value in the case of olive and chia oils (R^2= 0.8844 and 0.9768, respectively) than that in the case of garden cress oil (R^2= 0.7325). The correlation between viscosity and peroxide value differs according to the oil type and to establish a good correlation we need a lot of data and experiments.

3.3. FTIR fingerprint of crude and thermally processed olive, chia, and garden cress oils

During the thermal exposure of vegetable oils are happening, hydrolysis and oxidation reactions lead to changes into their functional groups which are visible within the Fourier transform infrared spectroscopy (Siddique, *et al*. 2015 and Jiang, *et al*. 2020).

Figure (2) illustrated the spectra of crude and heated oils investigated at different temperatures for 2 hours. The spectra are overlapping, but in some specific points are noticeable differences of absorption. As it can be observed, for both crude and heated oils of olives, chia seeds, and garden cress seeds strong absorption peaks are visible at 2924 cm^{-1} (asymmetric –C-H(-CH2) stretching), 2855 cm^{-1} (symmetric –C-H(-CH2) stretching), and at 1744 cm^{-1} (ester carbonyl stretching, -C=O) (Jiang, *et al*. 2020 and Saeed & Naz 2019). Little decrease in absorption at 2924, and 2855 cm^{-1} was observed in the case of olive and garden cress oils. While, a little decrease was observed in the absorption at 1744 cm^{-1} in the case of chia oil. These indicated a change in the structure of the investigated oils. Moreover, accepting the olive oil, *cis* double bonds stretching was visible at 3010 cm^{-1} for chia and garden cress oils, a fact that can be attributed to the very low percentage of polyunsaturated fatty acids in olive oil. It can be noticed a decrease in absorption at this wavenumber in the case of chia oil, while an increase was noticed in the case of garden cress and olive oils.

The vibration visible at 3200 – 3500 cm^{-1} in the case of chia and garden cress oils indicated the presence of peroxides produced during thermal treatment, while it is disappear in olive oil. Also, a peak at 1672 cm^{-1} was noticed in olive oil indicated the presence of free fatty acids.

According to Srivastava and Semwal (2015), at 962 cm^{-1} can be observed *trans* olefins bending. In Figure (2), it can be noticed a slight difference at 962 cm^{-1} between spectra for chia and garden cress oils, indicated formation of *trans* fatty acids in these oils. While it was not observed in the case of olive oil. This observation might be explained by gradually *cis* to *trans* isomerization of fatty acids during the thermal treatment (Siddique, *et al.* 2015 and Mitrea, *et al*. 2022).

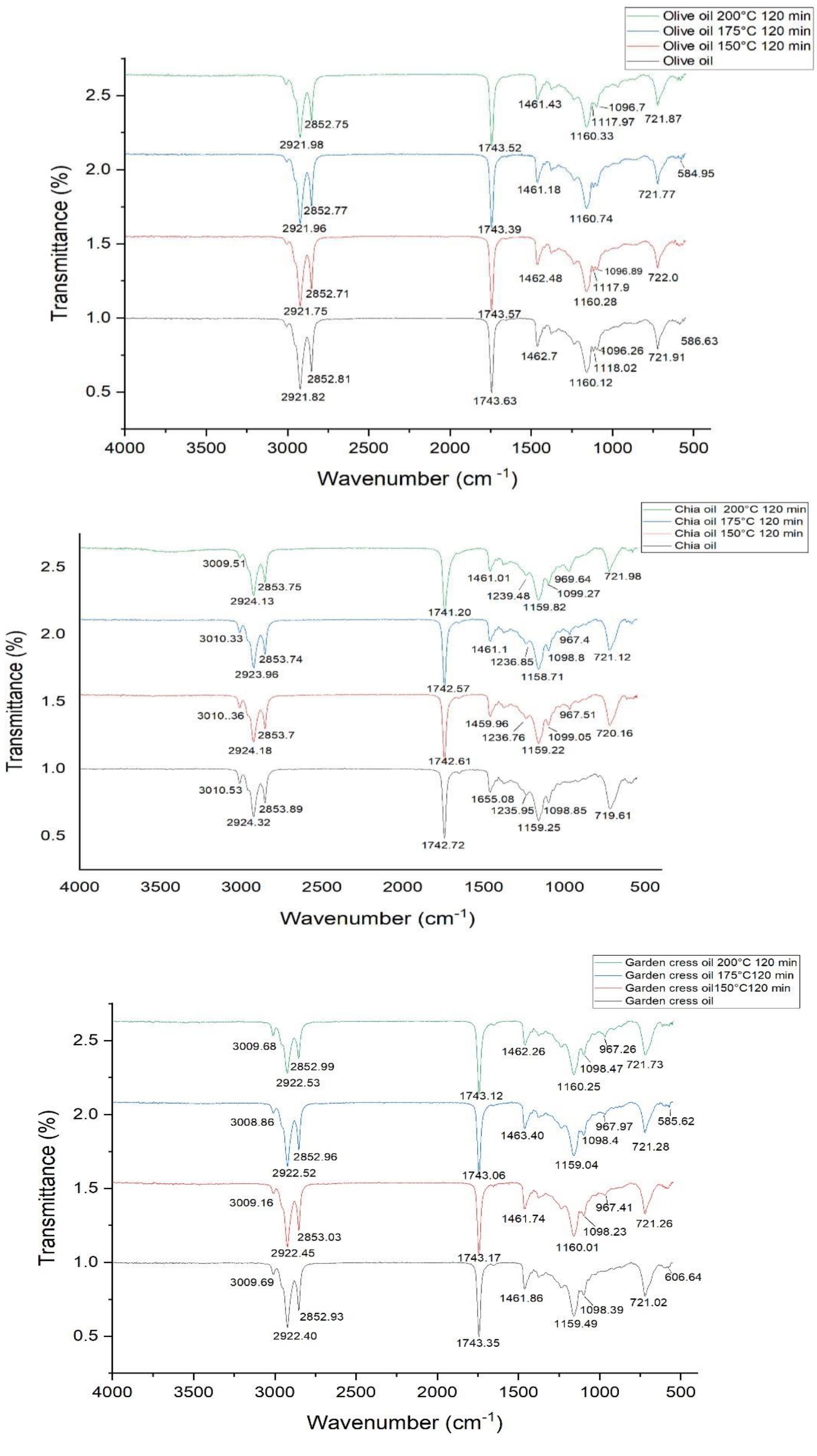

Figure 2. FTIR fingerprint of crude and thermally processed olive, chia, and garden cress oils at 150, 175, and 200 °C for 2 hours

4. Conclusion

Olive, chia and garden cress oils are directly affected by heat treatment. From the obtained results it can be noticed an important changes considering the peroxide value, rheological properties and functional groups of thermally treated oils. The thermal treatment induced oxidation, hydrolysis, and *trans* isomerization of the unsaturated fatty acids which were visible under the FTIR fingerprint. The modifications in the fatty acids profile after heat treatment increased viscosity of the tested oils. The rheological properties and FTIR spectra can be used as good techniques to estimate the quality of heated oils.

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Declaration of competing interest

All authors declare that there is no conflict of interests.

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