MICROWAVE HEATING EFFECT ON THE OXIDATION AND QUALITY OF COLD-PRESSED SUNFLOWER OIL

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Abstract

Sunflower oil is widely used in the human diet, primarily due to its constituent components and its applications in producing functional products. The use of microwave ovens in food preparation has become widespread in recent years due to certain advantages that include saving time, energy and convenience. As a result of heat treatment of oils, various chemical reactions can occur, leading to the formation of products that may indicate deterioration or a reduction in quality. For this purpose, untreated oil (not subjected to heating) and oil heated in a microwave oven for varying durations (ranging from 4 to 20 minutes) were analyzed in this study. The quality assessment of cold-pressed sunflower oil was conducted using standard official methods to measure peroxide, acid, and iodine values. The progression of oil oxidation was tracked by analyzing conjugated dienes and conjugated trienes through UV-spectroscopy (method AOCS Ch 5-91). Additionally, considering the theoretical link between microwave heating and changes in fatty acid composition, the fatty acid profile of the oil was determined according to MKC EN ISO 12966-4:2015. The results indicate a notable shift in the peroxide value (PV). Specifically, after heating the oil for 4 minutes, the PV exceeds the permitted limits. Furthermore, a decrease in the iodine value is observed with increasing heating time, while no significant changes are noted in the acid value. Additionally, changes in the content of fatty acids are observed in coldpressed sunflower oil. At specific wavelengths (232 and 268 nm), conjugated dienes and trienes were detected as oxidation products, i.e. the extinction coefficients (K_{232}) and (K_{268}) fell within the ranges of 3,72-4,16 and 1,25-2,56, respectively. These findings lead to the conclusion that microwave heating induces changes in the quality of cold-pressed sunflower oil. To maintain its quality, an appropriate choice of heat treatment is required.

Keywords: food quality, sunflower oil, microwave heating, products of oxidation

1. Introduction

Sunflower oil is known for its delightful taste and aroma, ease of refinement, stability, and most notably, its high content of linoleic acid, an essential fatty acid. It is transparent, with a golden or light-yellow color, when stored it does not form sediment, it has a faint smell of sunflower seeds. The unrefined sunflower oil has a dark yellow color, has a strong specific smell, and when stored it forms sediment (Oroian & Leahu, 2014). Sunflower oil is mostly used in the food industry as an edible oil, and then for the production of margarine, and mayonnaise (Jankuloska & Pavlovska, 2015), but it is also a good raw material for hydrogenation because it gives hydrogenated fat with a full white color, without any special smell and taste. Because of the components that sunflower oil contains, it can also be considered a functional food that has a significant benefit on human health (Bramley et al., 2000; Comlik & Pokory, 2000; Shahidi, 2005; Zingg, 2007; Makała, 2015).

Cold-pressed oils are obtained without chemical processing and accordingly, their nutritional and chemical composition differs from refined oils. Due to their production process, cold-pressed oils are highly susceptible to undesirable changes, including chemical reactions, enzymatic activity, and microbiological processes that lead to spoilage (Zhang et al., 2010). Under any conditions, degradation leads to the creation of degradation products that

compromise the sensory qualities of the oil, resulting in unpleasant smells and tastes. It is crucial to emphasize the potential harm to consumer health. Since all oils contain more or less unsaturated fatty acids, oxidation is an undesirable series of chemical reactions in oil that degrades its quality and value. Oxidation acts on the reactants (unsaturated lipids) and under the action of oxygen, primary products (peroxides) are obtained which further, while the reaction is ongoing, create secondary products (conjugated dienes, ketones, and aldehydes) (Dimić & Turkulov, 2000; Гулабоски, 2014). The sustainability of the oils is perceived through oxidative stability. Knowledge of sustainability is very important how in determining in advance the time for which these products can be stored without significant changes in quality (Dimić et al., 2004; Dimić, 2005).

Oil quality can be managed by monitoring quality parameters. The quality of the oils is defined in the Rulebook on the requirements regarding the quality of edible vegetable oils and fats of vegetable origin, margarine, mayonnaise, and related products (Official Gazette of the Republic of Macedonia, no.127, 2012). The peroxide value is a characteristic parameter and is used as an indicator of the primary oxidation of the oil (Talal et al., 2013). It shows the amount of hydroperoxides, as the primary product of autoxidation. Hydroperoxides have no taste or smell but are unstable and easily convert into other products such as aldehydes that have a strong and unpleasant smell and taste. The iodine value depends on the nature of the oil, the variety and hybrid of the raw material from which the oil is derived, the climatic conditions, the quality of the soil, etc (Karlović et al., 1996). The iodine value provides insight into the degree of unsaturation in fatty acids. The acid value determines the content of free fatty acids and is used as one of the parameters for determining the oil quality. Acidity can also be expressed as % free fatty acids (Павловска & Jaнкулоска, 2017). In the oxidation reaction after the peroxides are created, converting the unconjugated double bonds present in the unsaturated lipids into conjugated double bonds occurs. Conjugated dienes and trienes are formed in the process of autoxidation by hydrogen peroxides of unsaturated fatty acids and their fragmentation products (Боску, 2011).

The culinary industry widely utilizes microwave ovens because of their numerous advantages, including time and energy efficiency, as well as their ease of use (Cerretani et al., 2009; Lukešová et al., 2009; Zhou et al., 2021; Pielak et al., 2022; Xu et al., 2023). According to Abbas Ali et al. (2013), microwave heating is considered efficient and represents an ideal method for food preparation compared to the conventional method, and the advantage is due to the shorter processing time of the lower processing temperature. The nutritional properties of microwaved foods, specifically in vegetable oils and fats, have been researched by several authors (Vieira et al., 2001; Dostalova et al., 2005; Abbas Ali et al., 2016; Javidipour et al., 2017).

According to the above, this study will monitor the changes that occur in sunflower oil during thermal treatment in the microwave for different time intervals.

2. Materials and methods

This paper determines the quality parameters for sunflower oil before heating and after heating in the microwave at different time intervals. For this research, the cold-pressed sunflower oil from the manufacturer "Agrofila Dooel" packed in a 1 L plastic bottle, purchased from a market in Veles, Republic of North Macedonia, was analyzed.

Cold-pressed oils were analyzed both in their untreated state and after being heat-treated in a microwave oven (Samsung, Triple Distribution System, 800 W). Samples of oil (10 g) in glass erlenmeyer flasks (with an opening diameter of 2.4 cm) were placed in the center of a 27 cm diameter rotating plate and heated at 800 W for 4, 8, 12, 16, and 20 minutes. Following the

literature data that oxidation processes occur when the oil is heated, the indicated time intervals of heating have been selected.

Peroxide value was analyzed according to ISO 3960:2001 (AOCS Cd 8b-90), acid value according to ISO 660:1996 (AOAC, 1999; AOCS Metod Ca 5a-40 and Cd 3a-63), while iodine value was determined according to ISO 3961:1996 (AOAC 993.20; AOCS Cd 1d-92).

The analysis of the fatty acid composition was determined by gas chromatography with a flame ionization FID detector, a Shimadzu GC 2010 Plus instrument with FID and an AOC-5000 autosampler, with a SUPELCO SP-2380 column, 60m; 0.25mm i.d., df = 0.2 μ m according to MKC EN ISO 12966-4:2015.

Chromatographic analysis conditions: injector temperature 260°C, detector temperature 290°C, oven temperature program 60°C, hold 1 min, 17°C/min to 168°C, hold 28 min; total time of the program 35.35 min; gas-helium, pressure - 254.8 kPA, linear velocity - 35.4 cm/s, total flow - 53.3 mL/min, column flow- 2.32 mL/min, purge flow - 3.0 mL/min , injection volume 0.4 μ L, split ratio 1:20.7.

AOCS Ch 5-91 standard method was used for the determination of conjugated dienes and trienes in the oils.

3. Results and Discussion

3.1 Peroxide value (PV): The results of the analysis of the peroxide values of cold sunflower oil heated in a microwave oven at a time of 0 minutes (control sample), 4, 8, 12, 16 and 20 minutes are presented in Figure 1.

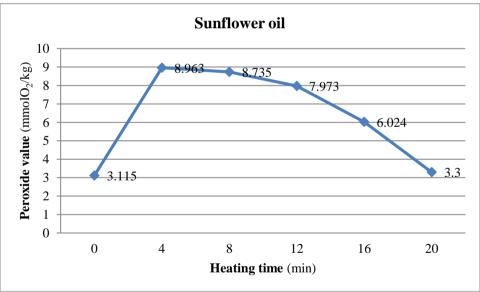


Figure 1. Peroxide values of sunflower oil

From the results obtained, it can be noted that the peroxide value of the control sample is 3,115 mmol O₂/kg. Comparing it with the maximum allowable concentration (7,5 mmol O₂/kg) of the Rulebook, we can conclude that the oil meets the quality requirements if consumed without heating. In the case of oil heated for 4 minutes, the peroxide value increases to 8,963 mmol O₂/kg and is above the maximum allowable concentration. If we continue heating the oil for 8 minutes, the peroxide value is reduced to reach a value of 8,735 mmol O₂/kg and up to 3,3 mmol O₂/kg when heating the oil for up to 20 minutes. The peroxide value is a parameter that indicates the oxidative stability of oils and fats stored under ambient conditions, as hydroperoxides are unstable at elevated temperatures. Microwave radiation promotes the rapid transformation of oils into secondary products, making the monitoring of peroxide value a standard analysis

during heating. However, more in-depth analysis is required to confirm the presence of these unstable components.

The rapid increase in peroxide value from 3,115 mmol O₂/kg in unheated oil to 8,963 mmol O₂/kg after 4 minutes of heating is likely due to the formation of hydroperoxides. With prolonged thermal treatment (beyond 4 minutes), these hydroperoxides decompose into secondary oxidation products, e.g. such as hexanal. Garcia-Martinez et al. (2009) point out that hexanal and heptanal are key components produced by microwave heating of oil and triacylglycerols rich in C18:2. Hexanal is known as a reliable indicator of fat and oil oxidation. Comparable results were obtained by Vieira & Regitano-D'Arce (2001) when analyzing canola oil. However, such a change in the peroxide values is probably also due to the fatty acids composition of the oil, especially due to the presence of C18:2 and therefore its value increases rapidly. According to Abbas Ali et al. (2016), the peroxide value of corn oil, heated in the microwave for varying durations, peaks at 8 minutes of heating before gradually declining. Hydroperoxides exhibit instability at temperatures exceeding 60°C, with their maximum concentration typically achieved shortly after 15 minutes of heating. Following this peak, the peroxide value begins to decrease with prolonged heating, as the rate of hydroperoxide decomposition surpasses that of hydroperoxide formation. Peroxide value reaches its highest

3.2 Acid value (AV): The acid values and free fatty acids (%), expressed as oleic acid, of sunflower oil heated in the microwave at different time intervals, are provided in figure 2 and 3.

value after 6 minutes of heating (Dostalova et al., 2005)

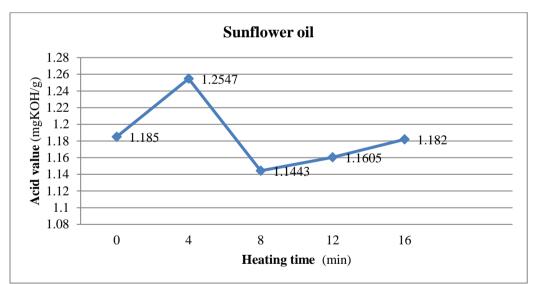


Figure 2. Acid values of sunflower oil

The acidity of the oils is included in one of the basic indicators of oil quality and that quantity is legally regulated. Thus, according to the Rulebook on fats and oils, free fatty acids (FFA) should be represented up to a maximum of 2% (maximum allowed values, MAV). Based on the results obtained, we can note that in all samples the % of the FFA is within the allowed values.

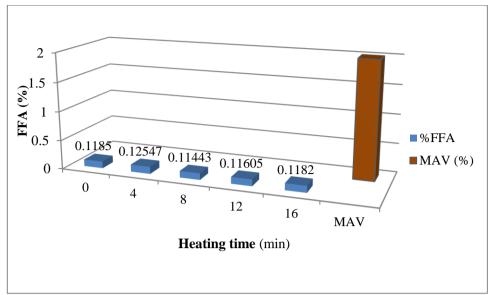


Figure 3. Free fatty acids (% FFA) of sunflower oil and maximum allowed values (MAV)

The free fatty acids present in the oils are the result of hydrolysis. There was a slight change between the acid value and the free fatty acids in the samples (figure 3). These results are consistent with research by both Tan et al.(2001) and Dostalova et al. (2005) who found that the change in acid values was negligible (less than 0,2 between the control sample and samples).

3.3 Iodine value (IV): The results of the analysis of the iodine values of sunflower oil are given in figure 4.

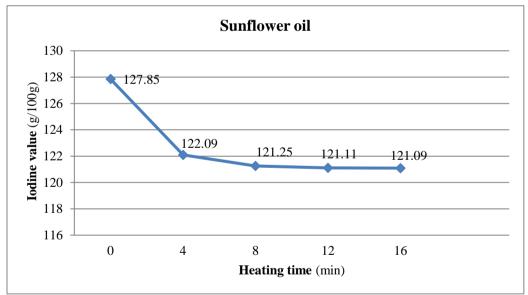


Figure 4. Iodine values of sunflower oil

As can be seen from the results in Figure 4, the iodine value of sunflower oil in the control sample is 127,85 g/100 g, which is by the Rulebook. As the heating time passes, it is noted that the iodine values gradually decrease, and the greatest decrease occurs when heating the oil for 4 minutes. Javidipour et al. (2017) found in their research that the iodine values in sunflower oil decreased the most when the oil was heated in a microwave oven for 3 to 6 minutes (Yoshida et al., 2003; Lukešova et al., 2009). The decrease in the iodine values continues, but it is inconspicuously small. Since the iodine value is associated with the unsaturation of the oil,

representing the number of double bonds present, the decrease observed during thermal treatment likely indicates a reduction in the oil's unsaturated bonds. These bonds are particularly susceptible to oxidation during heating, thus accounting for the decrease in iodine value in the samples. The presence of saturated and unsaturated fatty acids in the oil has an impact on the iodine value (Abbas Ali et al., 2016; Arifoğlu & Öğütcü, 2019).

3.4 Fatty acids composition: The results obtained from the analysis of fatty acids in sunflower oil are given in table 1.

Cold-pressed sunflower oil					
Fatty acids (% of	Heating time (min)				
total fatty acids)	0	4	8	12	16
C8:0	0,01	0,01	0,01	0,02	0,03
C10:0	0,01	n.d	n.d	0,04	n.d
C14:0	0,30	0,10	0,10	0,10	0,10
C16:0	6,10	6,10	6,40	6,20	6,20
C18:0	3,30	3,30	3,30	3,30	3,30
C20:0	0,26	0,28	0,28	0,28	0,28
C16:1	0,11	0,10	0,18	0,11	0,11
C18:1	35,30	35,20	35,20	35,70	35,60
C18:2	52,10	51,90	51,85	51,60	51,35
C18:3	0,27	0,26	0,25	0,24	0,24

 Table 1. Fatty acids composition of sunflower oil at different heating times

 Cold-pressed sunflower oil

 $n.d-not\ detected$

From the results obtained, we can note that microwave heating has an impact on the content of linoleic acid C18:2 and linolenic acid C18:3 in sunflower oil, the concentration of which decreases as the heating time of the oil increases. There is a slight decrease in the content of C18:3 acid as a result of microwave heating which is by the research of Javidipour et al. (2017). The increase in the content of C18:1 is noticeable and its content is highest when the oil is heated for 12 minutes. According to Anjum et al. (2006), this increase in C18:1 is associated with a decrease in C18:3 content. Concerning C18:3, its content is observed to increase as the oil is heated for an extended duration. These results are consistent with the results of Anjum et al. (2006), which noted that upon 15 minutes of microwave heating of sunflower seed, the C18:3 content increases, while the C18:1 content decreases. In C16:1, an increase in the content was observed at 8 minutes of heating. Comparable results were obtained by Javidipour et al. (2017). During the heating of the oil in the microwave oven, there is a slight increase in the values of saturated fatty acids C8:0 and C10:0, while C18:0 remains unaffected by microwave heating. In C16:0, there is a slight increase in its value, especially after 8 minutes of heating, as was obtained during the research of Javidipour et al. (2017).

After comparing the results with the Rulebook on fats and oils, it can be concluded that all fatty acids present in sunflower oil are within the allowed limits, except for C8:0 and C10:0, which should not be detected (Official Gazette of the Republic of Macedonia, no.127, 2012). In our samples, however, these two acids were detected in very small percentages.

3.5 Conjugated dienes and trienes: Results for the determination of conjugated dienes and trienes in sunflower oil heated at various durations are presented in table 2.

Tuble 2. Specific extinction at 252 and 200 million sumples					
Cold-pressed sunflower oil					
Heating time (min)	Specific extinction K ₂₃₂	Specific extinction K268			
0	3,85	1,25			
4	3,99	1,43			
8	4,16	1,71			
12	3,72	2,49			
16	3,76	2,56			

Table 2. Specific extinction at 232 and 268 nm of sunflower oil samples

During oxidation, conjugated compounds, and primary and secondary oxidation products occur, and determining the absorption maximum is important for defining the extent of the changes. From the results in Table 2, it is noted that the absorption maximum at a wavelength of 232 nm, indicates the presence of hydroperoxides, conjugated dienes, or primary oxidation products. Given that C18:2 is highly prevalent in sunflower oil, it can be noted that these maxima are also the result of the above. Absorption maximum at 268 nm and 270 nm gives the content of unsaturated carbonyl compounds, conjugated trienes, and secondary oxidation products arising from hydroperoxides (Dimić, 2005). Accordingly, the oxidation of polyunsaturated fatty acids is always followed by changes in absorption in the UV area. The specific absorption at 232 nm as a measure of the primary oxidation products always shows a tendency to increase with greater oxidation of fats and oils. Thus, the greater the absorption values at 232 nm and 270 nm, the oils are oxidized to a greater extent. During advanced oxidation, the primary products, i.e., conjugated dienes, will be converted into secondary products, which would lead to a decrease in absorption (Гулабоски, 2014).

From the results in Table 2, there is a significant increase in the values for K232 when heating the oil from 0 to 8 minutes. Similar results were obtained by the authors Kiralan & Ramadan (2016) when heating canola, saffron, and sesame oil. From the results obtained in Table 2, a constant increase in the value of K268 is observed (from 1,25 to 2,56). The content of conjugated trienes in hazelnut oil, sunflower oil, and soybean oil increases with 9 minutes of heating (Javidipour et al., 2017).

In sunflower oil, it is noted that there is increased content of trienes, and it is most likely due to the high content of C18:2. The values increase and reach a maximum value by increasing the duration of the microwave heating and the effect of the microwave treatment is greater in contrast to the conventional heating in vegetable edible oils (Albi et al., 1997). Dandjouma et al. (2006), confirm that the extinction coefficient in oil of *Canarium schweinfurthii* Eng. increases rapidly, according to the increase in the heating time in a microwave oven. Vieira & Regitano-Darce (2001) observed that absorption at 232 nm in canola oil, corn and soybean oils increases significantly after 12 minutes of heating, while absorption at 270 nm increases with 4 minutes of heating. Lukesova et al. (2009), observed that the peroxide values, conjugated dienes and trienes in corn oil, and sunflower oil increased as microwave heating progressed. Soybean oil and sunflower oil with higher C18:2 content show higher initial and final content of conjugated dienes, unlike olive oil and hazelnut oil (Javidipour et al., 2017).

4. Conclusions

Sunflower oil is widely used in the diet of humans, but it is also used as a raw material for obtaining a variety of products. When treating sunflower oil, special care should be taken because various thermal treatments contribute to the loss or reduction of important components and the creation of harmful components. In this study, it was shown that microwave heating leads to a change in the quality of the oil shown trough the peroxide value, the creation of conjugated dienes and trienes as a result of oxidation, and a change in the fatty acid composition especially of unsaturated fatty acids. Considering the conveniences and advantages offered by microwave food treatment as a widely used treatment by consumers, to preserve quality, the timing of thermal treatment of sunflower oil should also be considered. Given that oxidation products, such as peroxides and conjugated dienes, show irregular variations, and trienes increase with *prolonged* heating times, to evaluate the quality of oils heated in a microwave oven, further consideration should be given to other products (e.g. hexanal) as indicators of the impact of microwave heating on the quality of vegetable oils, which will be our subject for further research.

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