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Changes in cold-pressed oil due to roasting of hazelnut kernels

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A B S T R A C T

Hazelnut oil is a high-quality nutritional product, especially the oil obtained by cold pressing. Cold-pressed oils generally retain the original chemical composition and nutritional value of the hazelnut kernel. Due to the presence of unsaturated fatty acids, hazelnut oil is subject to oxidative and thermo-oxidative changes depending on whether the oil or kernels have been subjected to inappropriate storage or heat treatment. In this study, the influence of roasting hazelnut kernels at 180 °C for 24 minutes on cold-pressed oil was investigated. Roasting hazelnut kernels caused an increase in acid value in cold-pressed oil, which could indicate hydrolytic changes in triacylglycerols. There was a decrease in the refractive index, which also indicates changes in triacylglycerols and fatty acids. Roasting the kernels had no effect on the content of water and volatile matter. No peroxides were detected either, regardless of whether the kernels were subjected to heat treatment. However, it is clear from the specific absorbance values that the roasting of the hazelnut kernels initiated oxidative processes, and the change in the UV/Vis spectrum clearly indicates that the heat treatment of the kernels led to a deterioration of the oil, possibly as a result of changes in unsaturated bonds. However, the quality parameters of the cold-pressed oil from heat-treated kernels, i.e., the water and volatile matter content as well as the acidity and peroxide values, are in accordance with the regulations, as the changes in these parameters were minor or non-existent.

Keywords: hazelnut oil, roasting, oil oxidation, *triacylglycerol* hydrolysis

И З В О Д

Лешниково уље је високо вредан прехрамбени производ, посебно ако је добијено хладним пресовањем, када задржава иницијални хемијски састав сировине и хранљиву вредност уз минимум промена. Због свог карактеристичног хемијског састава, посебно доминантног присуства незасићених масних киселина, уље може да претрпи оксидативне и термо-оксидативне промене током неадекватног складиштења уља или језгара лешника или услед њиховог излагања повишеним температурама. У овом раду испитан је утицај термичке обраде језгара лешника на 180 °C у трајању од 24 min на квалитет хладно пресованог уља. Печење лешника је утицало на повећање киселинског броја хладно пресованог уља, што је индикатор хидролитичких промена триацилглицерола. Дошло је до смањења индекса рефракције, што такође указује на промене у триацилглицеролима и масним киселинама. Термичка обрада није утицала на садржај воде и испарљивих материја у уљу. Нису откривени пероксиди у уљима независно од излагања језгра лешника термичком третману. Међутим, на основу промена специфичних апсорбанција јасно је да је печење лешника иницирало оксидативне процесе и промена UV/Vis спектра јасно указује да је примењени термички третман језгара лешника узроковао погоршање квалитета хладно пресованог уља, као могућа последица промена код незасићених веза. Међутим, параметри квалитета хладно пресованог уља из термички третираних језгара: садржај воде и испарљивих материја, киселински и пероксидни број у складу су са прописима, јер су промене ових параметара биле минорне или нису ни забележене .

Кључне речи: лешниково уље, печење језгара, окидација уља, хидролиза триглицерида

1. Introduction

The hazelnut (*Corylus avellana* L.) is cultivated on a large scale in various countries due to the high demand for its nutritionally valuable kernels with their characteristic unique taste. The total area under hazelnut cultivation worldwide reached 1 million hectares in 2020, and the largest hazelnut producer is Turkey, which supplies almost 70% of the world's hazelnut production with 734,000 hectares, followed by Italy with 80,000 hectares, Azerbaijan with 45,000 hectares, and Chile and Iran with 24,000 hectares each (FAO, 2022). The hazelnut and its by-products are a good source of nutrients and bioactive compounds, which have almost the same chemical composition in different regions, but their content varies slightly (Zhao et al, 2023). Hazelnut kernels contain macro and micronutrients, proteins, oil, essential amino and fatty acids, minerals and vitamins. Using hazelnut in diet contributes to a better health status because of amino acid composition (the presence of a large amount of arginine), the specific composition of fatty acids and minor components: β-sitosterol, tocopherols (vitamin E), vitamin B6,quercetin, catechin, myricetin K, Ca, Mg, P, Se, etc. (Rondanelli et al, 2023; Solar and Stampar, 2011).

Only 10 % of the total hazelnut production is used for direct consumption in the form of fresh or roasted kernels. Most of the hazelnuts produced, around 70%, are used for chocolate and around 20% for ice cream and pastries (Guiné and Correi, 2020); its kernels can be used whole, crushed or processed into paste. In addition to the kernels, hazelnut oil is particularly interesting for use. The oil content of hazelnuts varies (Şahin et al., 2022; Müller et al., 2020), depending on the geographical location, variety and cultivation method, but the average values of commercial varieties are around 65% (Cittadini et al., 2022). Hazelnut oil can be obtained by pressing or extraction using chemical and physical techniques, with priority given to cold-pressed extraction due to nutrient preservation. Hazelnut oil has a golden yellow color and a very pleasant taste. Oleic acid is a dominant fatty acid (approx. 80% of total fatty acid content), which, together with tocols, makes hazelnut oil more resistant to oxidation than some other vegetable oils. Hazelnut oil is used in confectionery, as salad dressing, in cooking and in pharmaceutical and cosmetic products (Guiné and Correi, 2020; Medel et al, 2009).

Although the roasting of hazelnuts contributes to the release of the desired aroma, it also causes significant physico-chemical changes. Due to the high content of unsaturated fatty acids, the fatty acid composition of hazelnut oil changes during heat treatment due to oxidation (Liu et al., 2022; Radovanović et al., 2021). The antioxidant activity and phenolic compounds of the nuts are reduced by heating (Özcan et al., 2018). There are a number of physico-chemical analyses that can be used to determine changes in oils caused by thermal treatment. Acid value (AV) and peroxide value (PV) are basic parameters of oil quality and are usually checked during kernel processing. The AV is an indicator of possible triacylglycerol hydrolysis and the release of fatty acids. The PV indicates oxidative damage to the oil, which in most cases is due to a change in the unsaturated bonds of the fatty acids and the triglycerides in which they are contained. Liu et al. (2022) measured the peroxide value, anisidine value, total peroxide value, polar compound content, fatty acid content and aldehyde content in hazelnut oil during thermal treatment, indicating that hazelnut oil is not suitable for high temperatures and long heating times. Based on the oil content, PV value and specific absorption, Özkan et al. (2016) determined the optimal conditions for roasting kernels for two hazelnut varieties: for 'Delisava' 114.24 °C for 27.21 min and for 'Kara Fındık' 123.43 °C for 22.12 min.

Optical methods such as refractometry and spectrophotometry also provide useful insight into oil changes. The refractive index (RI) is a parameter for oil identification and depends on the saturation, cis-trans configuration and oxidation of fatty acids. The increased values of specific absorbances of the oil measured at 232 nm and 270 nm indicates primary and secondary oxidation products, respectively. UV-Vis spectrophotometric measurements are very useful for understanding changes in oil and, coupled with chemometrics, they were developed for authentication of edible oils (Jiang et al, 2015; Liu et al., 2013) or for determination of AV (Zhang et al, 2015). Ok (2017) examined olive oil adulteration by low-field NMR relaxometry and UV-Vis spectroscopy. Radovanović et al. (2021) showed changes in the UV part of the spectrum of cold-pressed oil from thermally treated hazelnut, which was in agreement with the increase in the peroxide value.

The aim of this study was to investigate the effect of roasting hazelnut kernels at 180 °C for 24 minutes on the properties of cold-pressed oil.

2. Materials and methods

2.1. Materials

The hazelnut kernels of the 'Tonda di Giffoni' variety were stored at -18 °C for 9 months prior to analysis. All chemicals were p.a. except n-hexane for spectrophotometric measurements (HPLC grade, Carlo Erba, France).

2.2. Determination of dry matter and crude oil content in hazelnut kernels

The dry matter (DW) content of ground hazelnut kernels was determined gravimetrically by drying the samples at 103 ± 2 °C to constant weight. Subsequently, the dried samples were used to determine the crude oil content by extraction in a Solvent extractor (Velp Scientifica-SER 148, Italy) with 70 mL of petroleum ether. The extraction parameters were: temperature 110 °C, immersion time 40 minutes, washing time 40 minutes. After recovery of the solvent, the solvent residue was evaporated in an oven (103 °C ± 2 °C, 30 minutes).

2.3. Thermal treatment and cold pressing of hazelnut kernels

The hazelnut kernels were thermally treated in an oven at 180 °C ± 2 °C for 24 minutes and then the thin skin was manually removed from the surface of the kernels.

The oil was obtained by the cold pressing of thermally treated and untreated hazelnut kernels with an electric screw press, power 650 W (OP 650W, Gorenje, Slovenia), with an output oil temperature of 35–37 °C (Figure 1). The oil yield (HO hazelnut oil or HOt hazelnut oil from thermally treated kernels) was calculated as the ratio of crude oil weight to the initial weight of the hazelnut kernels. The cake yield (HC hazelnut cake or HCt hazelnut cake from thermally treated kernels) was calculated as the ratio of cake weight to the initial weight of the hazelnut kernel. The cold-pressed oils were left overnight at 4 °C. The oil was separated from the sediment by decanting and then analyzed.

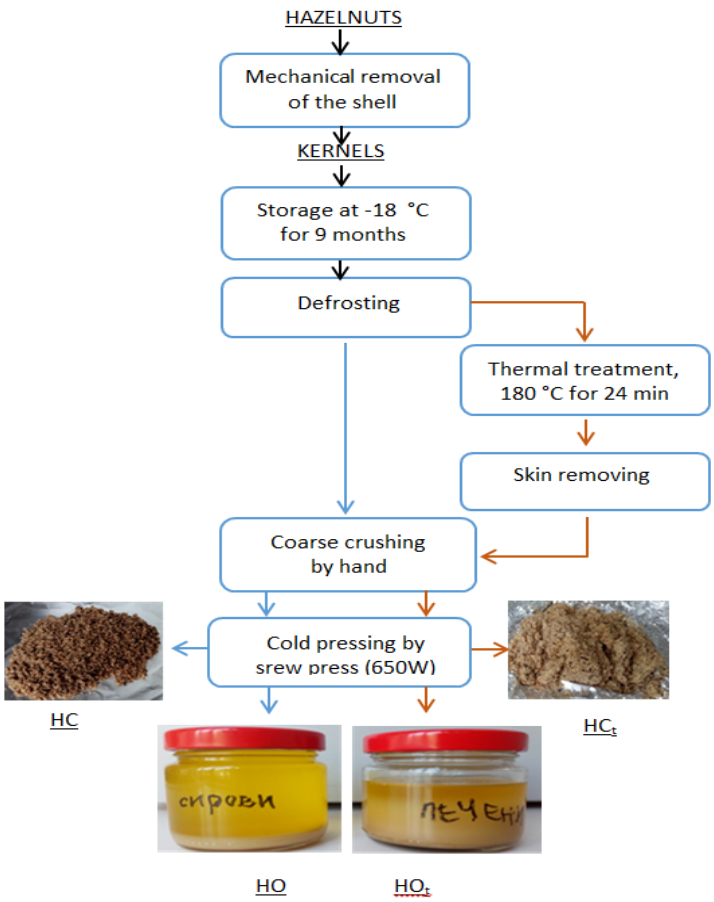

Figure 1. Obtaining cold-pressed hazelnut oil without heat treatment and after heat treatment of the kernel

2.4. Determination of moisture and volatile matter content and refractive index in oil

The oil samples were dried at 103 °C ± 2 °C until complete elimination of water and volatile substances to a constant mass of dry residue (ISO 662, 1980; Dimić and Turkulov, 2000). The result was expressed as moisture and volatile matter (MVM) content in % (w/w).

The dried oil samples were used to determine the refractive index (RI) according to a standard method (ISO 6320, 1995; Dimić and Turkulov, 2000) with an Abbe refractometer (A. KRUSS, Germany), range 1.3000–1.7000 ± 0.0001, and a monochromatic Na light (589.6 nm) at 28 °C.

2.5. Determination of acid and peroxide value

The acid value (AV) was determined volumetrically by neutralization of free fatty acids with 0.1 M ethanolic KOH solution with phenolphthalein as an indicator (Dimić and Turkulov, 2000). The AV was calculated according to equation (1) and expressed as mg KOH per g oil:

$$AV\ (mg\ KOH/g) = \frac{56.1 \cdot V \cdot c}{m} \tag{1}$$

where V is volume of titrant in mL, c is exact concentration of ethanolic solution of KOH and m-weight of oil in g.

The peroxide value (PV) was determined according to the modified Wheeler method (Dimić and Turkulov, 2000). The samples were titrated with a 0.01 M aqueous solution of $Na_2S_2O_3$ with starch solution as an indicator. The peroxide value is the amount of active oxygen in the lipids corresponding to the amount of iodine released from KI. The PV was calculated according to equation (2) and expressed in mmol per kg of oil.

$$PV\ (mmol/kg) = \frac{(V_1 - V_0) \cdot f \cdot 5}{m} \tag{2}$$

where V_1 is volume of titrant used for sample, in mL; V_0 volume of titrant used for blank; f is factor for exact concentration of water solution of $Na_2S_2O_3$ and m-weight of oil in g.

2.6. Determination of specific absorbances and recording of the UV-Vis spectrum

The specific absorbances of the oil solution in n-hexane were measured in the UV part of the spectrum at 232 nm and 270 nm for a concentration of 1 g per 100 ml (ISO 3656, 1989; Dimić and Turkulov, 2000) and were calculated according to equation:

$$K_\lambda = \frac{A_{(\lambda)}}{c} \tag{3}$$

For the recording of UV-Vis spectra, oil samples were prepared by dissolving oil in n-hexane (1:5, v/v).

The spectra were recorded in the wavelength range from 200 nm to 800 nm with a data interval of 1 nm and a scan rate of 600 nm/min. The Cary WinUV software was used. All spectrophotometric measurements were performed on a Cary 300 spectrophotometer (Agilent, USA).

The results were shown as the mean value of three measurements with standard deviation by analysis in Microsoft Excel software.

3. Results and discussion

The hazelnut kernels were thawed after 9 months of storage at -18 °C and then divided into two parts, one of which was subjected to heat treatment. The oil from the hazelnuts was obtained by cold pressing. Roasting the hazelnut kernels affected the color of the cold-pressed oil, resulting in a darker color (Figure 1). On the other hand, the cake was lighter in color, which can be attributed to the fact that the skin of the roasted kernels was removed. The high DW content could indicate adequate drying treatment after harvesting (Table 1) before freezing. After harvesting, the moisture content must be reduced to about 4–6 %, with variations noted by different authors (Vrtodušić et al., 2022). Reducing moisture content is crucial to preventing chemical, enzymatic or microbiological food spoilage. The crude oil content was about 58 % per DW (Table 1). Luciani et al. (2020) detected 62.4 % oil in 'Tonda di Giffoni' kernels, Müller et al. (2020) determined about 62.7 % of the fresh weight, while Cittadini et al. (2020) found about 70 % oil per DW in the same variety. In addition to the hazelnut variety, the chemical composition of hazelnut kernels also depends on the growing conditions, the degree of ripeness of the harvest, drying and storage conditions, and the oil content also depends on the extraction method.

Table 1.
Hazelnuzt kernel properties and thermal treatment effect on the yield of cold-pressed oil and cake

	Hazelnut kernels		Untreated kernels	Thermal treated kernels
DW, %	96.04 ± 0.06	Oil yield, %	47.66	42.55
Crude oil, % (w/DW)	57.75 ± 2.16	Cake yield, %	33.93	55.21

The thermal treatment (180 °C for 24 min) of hazelnut kernels led to a reduction in the yield of cold-pressed oil (Table1). There were lower pressing losses (100%-oil yield-cake yield) by cold pressing roasted kernels. With thermally treated kernels, a considerable amount of oil remains in the cake. Özcan et al. (2018) found that a 20-minute thermal treatment at 130 °C led to an increase in oil yield during extraction. Özkan et al. (2016) showed that the oil yield changed with increasing temperature and roasting time. In the temperature range of 100 to 150 °C, the oil yield increased, but a further increase had no effect on the oil yield obtained by extraction (Özkan et al, 2016).

The applied heat treatment led to certain changes in the cold-pressed hazelnut oil (Table 2). An increase in AV may indicate hydrolytic changes in triacylglycerols and the release of free fatty acids. However, the changes were minor and AV was well below the prescribed maximum value: 4 mg KOH/g for unrefined oils (Official Gazette (2013). The AV and PV were higher in the oils from the roasted hazelnuts (Manzo, 2017). Thermal treatment caused a reduction in RI (Table 2), indicating possible thermo-oxidative changes. It is known that when oil is thermally treated, the RI decreases due to changes in the basic structure of fatty acids, which also correlates with a decrease in iodine value; the quality of the oil was reduced by thermal treatment (Dimić and Turkulov, 2000). The RI value is a characteristic used for identifying the oil. It depends on the degree of unsaturation, the ratio of the cis/trans configuration of the fatty acids and the degree of oxidation. Hazelnut oil contains about 75% monounsaturated acids and, due to its physical and chemical properties, has an RI of 1.466 to 1.468 at a temperature of 25 °C (Sahin et al., 2022), and an RI of 1.462–1.463 at a temperature of 40 °C (Dimić, 2005).

Table 2.
Changes in cold-pressed oil due to the thermal treatment of hazelnut kernels

Cold-pressed oil from kernels	% *MVM*	*AV* (mg KOH/g)	*PV* (mmol O_2/kg)	$n_{D,}$ 28	K_{232}	K_{270}
Untreated	0.04	0.27 ± 0.07	0	1.4655 ± 0.000	1.03 ± 0.04	0.11 ± 0.06
Thermal treated	0.04	0.46 ± 0.02	0	1.4644 ± 0.000	1.12 ± 0.01	0.14 ± 0.05

MVM – moisture content and volatile matter; *AV* – acid value; *PV* – peroxide value; n_D 28 – refractive index measured at 28 °C; K_{232} and K_{270} – specific absorbances at 232 nm and 270 nm, respectively.

The specific absorbance of the oil at 232 nm increased after the thermal treatment of kernels (Table 2), indicating oxidative changes. There was no significant change in specific absorbances at 270 nm. Increased specific absorbance values at 232 nm indicate the formation of primary oxidation products, peroxides and hydroperoxides. Increased absorbance values at 270 nm indicate the formation of secondary diene and triene oxidation products. An increase in the value of K_{232} in relation to K_{270} indicates that the oxidative processes have only just begun and that secondary oxidation products have not formed or have formed only in negligible quantities.

There was no effect of kernel thermal treatment on the content of moisture and volatile materials in the oil. This quality parameter was in accordance with Official Gazette (2013), which allows a maximum MVM content of 0.20% for cold-pressed edible oils. The presence of peroxides was also not detected by PV determination, which may be a consequence of the insufficient sensitivity of the method to the minor changes that occurred, indicated by specific absorbance values, changes in RI, but also changes in the appearance of the characteristic UV/Vis spectrum (Figure 2). Radovanović et al. (2021) also found the absence of peroxides in cold-pressed oil from hazelnut kernels thermally treated at 105 °C for 1 hour.

Certain peaks in the visible light range (400 nm – 500 nm) are due to the presence of carotenoids. It is clear that there are no significant changes in this part of the spectrum, but on the other hand there are very pronounced changes in the appearance of the spectrum in the UV range. The hexane solution of unroasted hazelnut oil had a characteristic peak at a wavelength of about 290 nm. This absorption peak is lost in oil from roasted hazelnuts and the oxidation that occurs can be observed.

Each oil is characterized by a specific appearance of the UV-Vis spectrum, such as a characteristic "fingerprint". The thermal treatment led to a disturbance of the natural composition of the oil, which changes the appearance of the spectrum (Figure 2).

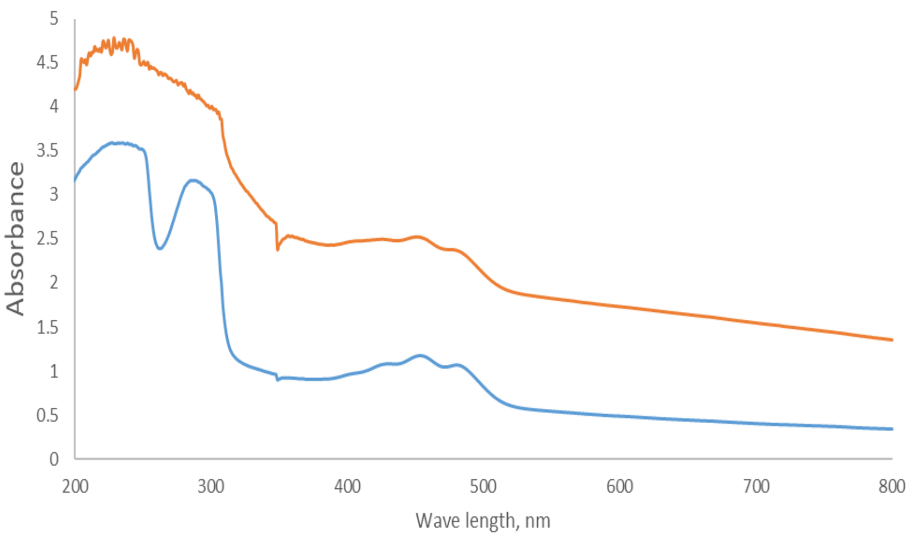

Figure 2. The effect of roasting the kernels at 180 °C for 24 minutes on the UV/Vis spectrum of cold-pressed hazelnut oil: untreated kernels (—) and thermally treated kernels (—)

The characteristic absorption maxima in this region are due to the presence of unsaturated bonds in fatty acids such as oleic acid and linoleic acid, which are primarily responsible for the broad absorption band with a maximum around 280 nm (Figueiredo et al., 2016). This indicates probable changes in the structure of the unsaturated fatty acids. Changes were detected in the same part of the spectrum of cold-pressed oil from thermally treated hazelnut kernels at 105 °C for 1 h after 4 months of storage (Radovanović et al., 2021).

4. Conclusions

Hazelnuts and hazelnut oil have a high nutritional value and can have positive effects on human health. However, thermal processing of hazelnut kernels or oil degrades their quality and often produces undesirable harmful products. The roasting of hazelnuts kernels at 180 °C for 24 minutes led to a possible hydrolytic degradation of triacylglycerol and an increase in the acid value. The changes in triacylglycerol and fatty acids were also reflected in a decrease in the refractive index. Thermal treatment had no effect on the water and volatile content of the oil (0.04% for untreated and thermally treated hazelnut oil) and on the peroxide value (0 mmol O_2/kg oil in both cases). The change in the UV/Vis spectrum clearly indicated that the thermal treatment of the kernels led to a disturbance of the native chemical composition of the oil, possibly as a result of changes in unsaturated bonds. The oxidative processes had only just begun and secondary oxidation products had not formed or only formed in negligible quantities. Regardless of all the changes observed and quantified in the oil as a result of the thermal treatment of hazelnut kernels, the oil obtained meets the prescribed quality requirements. The content of water and volatile components, and the acid and peroxide value comply with the regulations.

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Declaration of competing interests

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