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Effects of Roasting on Hazelnut Lipids

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The effect of roasting on some nutritional characteristics of hazelnut lipidic fraction was investigated. Hazelnuts (*Corylus avellana* L.) were submitted to several different thermal treatments, comprising different temperatures (125−200 °C) and times of exposure (5, 15, and 30 min) and analyzed for their moisture and crude fat. Raw and roasted hazelnuts were also analyzed for their compositions in phytosterols and fatty acids (including trans isomers) by GC-FID, triacylglycerols by HPLC-ELSD, and tocopherols and tocotrienols by HPLC-DAD/fluorescence spectroscopy. Minor changes occurred in the fatty acid and triacylglycerol compositions. As temperatures and roasting periods increased, generally, a modest increase of oleic and saturated fatty acids and a decrease of linoleic acid, expressed as relative percentages, occurred. Similarly, an increase of triacylglycerols containing oleic acid moieties and a decrease of those containing linoleic acid moieties were found in the roasted samples. Roasting caused a modest decrease of the beneficial phytosterols (maximum 14.4%) and vitamin E homologues (maximum 10.0%) and a negligible increase of the trans fatty acids.

KEYWORDS: Hazelnut; roasting; fatty acids; triacylglycerols; sterols; vitamin E homologues

INTRODUCTION

Hazelnuts (*Corylus avellena* L.) are widely consumed all over the world, and production in 2004 achieved almost 700 000 tons (*1*). Turkey dominates world production, producing >70% of the world's hazelnuts, followed by Italy (12%), the United States (6%), and Spain (2%) (*1*). Hazelnuts represent a product of great economic importance for these producing countries and the industries that use hazelnuts. They are used as an ingredient in the production of a great variety of manufactured food products including bakery goods, cereals, snacks, ice creams, various dessert formulations, and mostly pastry and chocolates (*2*, *3*). Only a small part (8−10%) of the annual hazelnut production is consumed as raw nuts (*2*). Frequently, they are submitted to thermal processing, mainly roasting, to obtain characteristic sensory or texture features. Besides extending the range of aromas, textures, and taste of hazelnuts, roasting is also performed to remove the pellicles (seed coats) of the kernels, inactivate enzymes, destroy microorganisms, and reduce water activity (*3*, *4*).

The thermal treatment of nuts during roasting inevitably leads to chemical changes mainly determined by the nut composition (moisture, fat, proteins, and carbohydrates) and by the temperature and extent of heating (*4*). Optimization of roasting conditions, by evaluating the kinetics of the process, moisture and drying characteristics, color, oxidative stability, sensory characteristics, and microstructural changes, has been extensively described in the literature (*5*−*11*). In contrast, studies regarding the changes on the chemical composition and the effects on the nutritional value of roasted hazelnuts are limited. Langourieux et al. (*12*) reported the influence of roasting parameters in hazelnut aroma compounds, and Alasalvar et al. (*13*) have compared the volatile compositions of raw and roasted hazelnuts. Ozdemir et al. (*3*) reported the effects of roasting on the amino acid composition, peroxide value, and the thiamin, riboflavin, and free fatty acid contents. Kirbaslar et al. (*14*) studied the roasting effects on the moisture, oil, protein, total carbohydrate, total sugar and starch, and fatty acid composition of hazelnuts. Although the lipid fraction is the major component of hazelnuts (∼60%), its behavior during roasting and subsequent effects on the nutritional value have been scarcely studied (*3*, *14*). This fraction, besides presenting a high content of unsaturated fatty acids, is also rich in important compounds with potential beneficial health effects such as phytosterols and vitamin E. Phytosterols are known for their ability to reduce blood cholesterol and, more recently, have also been reported to have anticancer and immune system modulating properties (*15*, *16*). Vitamin E, a family of eight lipid-soluble tocochromanols (α-, β-, γ-, and δ-tocopherols and tocotrienols), is believed to be involved in diverse physiological and biochemical functions, mainly due to its action as an antioxidant but also by acting as a membrane stabilizer (*17*). On the other hand,

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trans fatty acids, naturally formed during roasting, are believed to increase the risk of cardiovascular disease, therefore being prejudicial to human health (*18*).

The objective of this work was to study the influence of roasting on the nutritional value of hazelnuts' lipidic fraction. For this purpose, fatty acid, with emphasis on trans fatty acids, triacylglycerols, phytosterols, tocopherols, and tocotrienols compositions of hazelnuts, raw and submitted to different roast intensities, were evaluated and compared.

MATERIALS AND METHODS

Samples. Raw shelled hazelnuts were supplied from an industry located in Mangualde, a city in the central region of Portugal. From the observation of the size and shape of the nuts, the sample was composed of a mixture of two or three cultivars. Nevertheless, care was taken that all of the samples analyzed were representative of the same initial batch (50 kg), which was homogeneous. Several roasting experiments were performed on a laboratory scale using a WCT blinder stove (Tuttlinger, Germany). In the industry, roasting generally is performed at 145 °C for 15 min, so this condition was reproduced at the laboratory scale. Besides these conditions, the following were also tested: 125 °C, 15 min; 125 °C, 30 min; 165 °C, 15 min; 165 °C, 30 min; 185 °C, 15 min; and 200 °C, 5 min.

Reagents and Standards. The FAME standards were all from Supelco (Bellefonte, PA). Sterol standards (cholestanol, cholesterol, campesterol, stigmasterol, β-sitosterol, β-sitostanol, and betulin) and the triacylglycerol standards (PPP, SSS, PoPoPo, OOO, LLL, LnLnLn, PLL, OLL, PPO, SOO, POL and POO, where P is palmitoyl, S is stearoyl, Po is palmitoleoyl, O is oleoyl, L is linoleoyl, and Ln is linolenoyl) were purchased from Sigma (St. Louis, MO). Tocopherols (α, β, γ, and δ) and tocotrienols (α, β, γ, and δ) were from Calbiochem (La Jolla, CA), and the internal standard, tocol, was from Matreya Inc. (Pleasant Gap, PA). BHT was obtained from Aldrich (Madrid, Spain).

n-Hexane, acetonitrile, and acetone were of HPLC grade from Merck (Darmstadt, Germany) and 1,4-dioxane was from Fluka (Madrid, Spain). All other reagents were of analytical grade from several suppliers.

Sample Preparation. The samples were chopped in a 643 MX home coffee mill (Moulinex, Madrid, Spain) to pass through a sieve of 0.7 mm, and their oil was extracted with light petroleum ether (bp 40−60 °C) in a Soxhlet apparatus (Büchi, Flawil, Switzerland). The residual solvent was removed by flushing with nitrogen. This extracted oil was used for the analysis of fatty acids, phytosterols, and triacylglycerols. Tocopherols and tocotrienols were analyzed directly on the chopped samples as described below.

Moisture and Crude Fat. To express the results on a dry basis, moisture was determined at 100 ± 2 °C (~3 g test sample) with an SMO 01 infrared moisture balance (Scaltec, Goettingen, Germany). Total fat contents were determined according to AOAC Official Methods (*19*).

Fatty Acids (FA). FA were determined by gas−liquid chromatography with flame ionization detection (GC-FID) as previously reported by Amaral et al. (*20*). Briefly, the extracted oil samples were hydrolyzed with boiling methanolic potassium hydroxide solution (11 g/L), and the FA released were converted to methyl esters using BF_3/MeOH and extracted with *n*-heptane. The fatty acid profile was analyzed with a Chrompack CP 9001 chromatograph (Chrompack, Middelburg, The Netherlands) equipped with a split−splitless injector and a Chrompack CP-9050 autosampler. The temperatures of the injector and detector were 230 and 270 °C, respectively. Separation was achieved on a 50 m × 0.25 mm i.d. fused silica capillary column coated with a 0.19 μm film of CP-Sil 88 (Chrompack). The carrier gas used was helium at an internal constant pressure of 120 kPa. The column temperature was 160 °C, for a 1 min hold, and then programmed to increase to 239 °C at a rate of 4 °C/min and then held for 10 min. The split ratio was 1:50, and the injected volume was 1.2 μL. The results are expressed in relative percentage of each fatty acid, calculated by internal normalization of the chromatographic peak areas.

Phytosterols. Phytosterols were analyzed by a GLC-FID methodology as reported elsewhere (*20*). The same equipment as described for the fatty acid analysis, with a 30 m × 0.25 mm i.d., 0.25 μm DB-5MS column (J&W Scientific, Folsom, CA), was used. The temperatures of the injector and the detector were both 320 °C. The column initial temperature was 250 °C, programmed to increase at a rate of 2 °C/min to 300 °C and then held for 12 min. The split ratio was 1:50, the injected volume was 1.5 μL, and the internal pressure of the carrier gas was 100 kPa. Quantification (milligrams per 100 g of hazelnut oil) was achieved by the internal standard method, using betulin as internal standard.

Triacylglycerols (TAG). TAG were determined by HPLC-ELSD based on the methodology reported by Amaral et al. (*21*). A 0.2 g oil sample was dissolved in 4.0 mL of acetone and homogenized by stirring. The mixture was filtered through a 0.22 μm disposable LC filter disk and analyzed with a Jasco HPLC (Jasco, Tokyo, Japan), equipped with a PU-1580 quaternary pump and a Jasco AS-950 automatic sampler with a 10 μL loop. Detection was performed with a model 75 evaporative light scattering detector (ELSD) (Sedere, Alfortville, France). The chromatographic separation of the compounds was achieved with a 250 × 4.6 mm i.d., 5 μm, Kromasil 100 C_{18} column (Teknokroma, Barcelona, Spain) operating at ambient temperature (~20 °C). The mobile phase used was a mixture of acetone/acetonitrile (70:30, v/v) (A) and acetone/acetonitrile (80:20, v/v) (B). Elution was performed at a solvent flow rate of 1 mL/min with a two-step gradient, starting with 0% B and changing to 100% B at 35 min, keeping these conditions during 15 min and then returning to the initial conditions. The ELSD was programmed with the following settings: evaporator temperature, 40 °C; air pressure, 3.5 bar; and photomultiplier sensitivity, 5. Data were analyzed using the Borwin-PDA Controller software (JMBS, France). Peaks were identified by taking into account the selectivities (α, relative retention time of each compound to OOO); identification was also supported by the literature, based on hazelnut oil mass spectrometry data (*22*, *23*). Quantification of the peaks was made by internal normalization, assuming that the detector response was similar for all compounds (*24*).

Tocopherols and Tocotrienols. The samples were prepared using the validated methodology reported by Amaral et al. (*25*). Tocol was used as internal standard, and BHT was used as an antioxidant. Samples were analyzed in a Jasco high-performance liquid chromatograph, equipped with a PU-980 pump, an AS-950 autosampler with a 10 μL loop, an MD-910 multiwavelength diode array detector (DAD), and an FP-920 programmable fluorescence detector, programmed at the excitation and emission wavelengths of 290 and 330 nm, respectively. Separation was performed on a 250 × 3 mm i.d. Inertsil 5 SI normal phase column (Varian, Middelburg, Netherlands) operating at ambient temperature. The mobile phase was a mixture of *n*-hexane/1,4-dioxane (95.5:4.5, v/v) in an isocratic program (flow rate, 0.7 mL/min). Quantification was made by fluorescence detection based on the internal standard method.

Statistical Analysis. Statistical analysis involved the performance of MANOVA, Hotelling T^2, and Student's *t* tests, using the Statistica for Windows statistical package (Statistica for Windows, StatSoft Inc., Tulsa, OK).

RESULTS AND DISCUSSION

Moisture and Total Fat Content. Moisture and total fat content (calculated on a dry basis) of raw and roasted hazelnuts are reported in **Table 1**. As expected, the higher the temperature and roasting time, the higher the loss of water. Although roasting resulted in higher amounts of oil extracted from hazelnuts, this increment was not statistically significant ($P > 0.05$). This increase was probably due to the improved lipid extractability caused by both higher damage in the membranes and higher amount of protein denaturation (*26*, *27*). The color development increased significantly, from pale yellow to brown, as roasting temperature increased. This color change is usually ascribed to the formation of browning substances as a result of Maillard-type nonenzymatic reactions between reducing sugars and free amino acids or amides (*27*).

FA Composition. Because roasting is usually carried out at high temperatures in the presence of oxygen, there is a high

Table 1. Effect of Roasting on Moisture, Crude Fat, and Color[a]

	moisture (%)	fat (%, dw)	color
raw	5.0 ± 0.4 e	68.3 ± 0.7 a	pale yellow
125 °C/15 min	1.9 ± 0.1 d	68.4 ± 1.6 a	pale yellow
125 °C/30 min	1.6 ± 0.0 c	70.2 ± 0.1 a	yellow
145 °C/15 min	1.6 ± 0.0 cd	70.0 ± 0.8 a	yellow/gold
165 °C/15 min	1.5 ± 0.0 bc	68.5 ± 0.7 a	yellow/gold
165 °C/30 min	1.2 ± 0.1 ab	70.7 ± 1.4 a	brown
185 °C/15 min	1.0 ± 0.1 a	70.8 ± 1.9 a	dark brown
200 °C/5 min	1.4 ± 0.1 bc	69.4 ± 0.8 a	brown

[a] Mean ± SD, $n = 3$. Data followed by different letters within each column are significantly different according to Student's t tests at $P < 0.05$. dw, dry weight.

probability that lipid oxidation occurs, thus decreasing the hazelnuts' nutritional value. Because the FA composition of the oil can be an indicator of its stability and is also very important in terms of its nutritional value, the FA compositions of raw and roasted hazelnuts were evaluated and compared. In the raw sample 14 fatty acids were detected, as previously reported (*20*). Oleic (ranging from 77.9 to 82.2%), linoleic (ranging from 9.8% to 13.8%), palmitic (ranging from 4.8 to 5.5%), and stearic (ranging from 1.7 to 2.3%) acids were the major fatty acids both before and after thermal processing. Changes in the fatty acid relative percentages were observed with increasing roasting temperatures and times of exposure (**Table 2**). In general, the relative levels of saturated fatty acids and of oleic acid were found to increase while that of the linoleic acid was found to decrease. It is known that the rate of fatty acid oxidation is higher as the number of double bonds increases, which explains the results obtained (*28*). Theoretically, linolenic acid would isomerize at a higher rate, being a good indicator of heat treatment (*29*). However, because its level in hazelnuts is very low (<0.1%), the occurring trans isomers are below the detection limit. However, two exceptions to the above-referred general behavior were found: when roasting was performed at 165 and 185 °C, for 15 min. At 185 °C roasting, the levels of oleic acid were lower and those of saturated and linoleic acids were higher than in the raw sample. Because hazelnuts contain much higher amounts of oleic than of linoleic acids, probably greater contents of oleic acid were affected at this elevated temperature, thus lowering its relative levels and, as a consequence, increasing saturated and polyunsaturated fatty acid percentages. The degradation rates of oleic and linoleic acids seem to be identical at 165 °C during 15 min of roasting, because the fatty acid composition of hazelnuts submitted to this treatment was identical to that of the raw sample. A similar behavior was already reported in a previous work by Crews et al. (*30*). The results obtained are also consistent with other previously published work because changes in the fatty acid profile have already been described in several foodstuffs subjected to thermal treatments (*28*, *31*−*33*). With respect to trans fatty acids, the roasted samples presented only a minor increase (from 0.02% in raw hazelnuts to a maximum of 0.07% in the roasted samples). Although some prejudicial health effects with regard to trans fatty acids intake (*18*) have been reported, other authors state that at current levels of intake, dietary trans fats pose no health problems and that more studies are necessary to associate these compounds with harmful effects to human health (*34*). Although the increase of trans fatty acids was statistically significant ($P < 0.05$), their presence in the roasted hazelnuts is of no concern to public health because they were found to exist in minor amounts (<0.1%), thus having a negligible impact on the nutritional value of hazelnuts.

Table 2. Effect of Roasting on Fatty Acid Composition[a]

	fatty acid (relative %)																			
	C14:0	C16:0	C16:1w7c	C17:0	C17:1w7	C18:0	C18:1w9t	C18:1w9c	C18:2w6cc	C20:0	C20:1w9	C18:3w3c	C22:0	C22:1w9	SAT	MUFA	PUFA
raw	0.03 ± 0.00 c	5.02 ± 0.01 c	0.19 ± 0.00 bc	0.19 ± 0.00 e	0.07 ± 0.00 e	1.89 ± 0.01 c	0.02 ± 0.00 a	79.57 ± 0.01 c	12.72 ± 0.01 f	0.18 ± 0.01 ab	0.16 ± 0.00 ab	0.08 ± 0.01 e	0.02 ± 0.00 b	0.03 ± 0.00 c	7.33	80.03	12.80
125 °C/15 min	0.04 ± 0.00 d	5.33 ± 0.01 f	0.20 ± 0.01 bd	0.20 ± 0.00 f	0.07 ± 0.00 e	2.18 ± 0.01 g	0.02 ± 0.00 b	81.06 ± 0.02 f	10.59 ± 0.02 c	0.19 ± 0.01 ab	0.16 ± 0.00 b	0.07 ± 0.00 c	0.02 ± 0.00 d	0.04 ± 0.00 d	7.95	81.55	10.66
125 °C/30 min	0.04 ± 0.00 e	4.76 ± 0.01 a	0.16 ± 0.01 a	0.16 ± 0.00 de	0.06 ± 0.01de	1.95 ± 0.00 d	0.07 ± 0.01 f	80.98 ± 0.01 e	11.47 ± 0.01 d	0.19 ± 0.02 a	0.17 ± 0.00 b	0.07 ± 0.00 c	0.02 ± 0.01 d	0.04 ± 0.01 c	7.11	81.49	11.54
145 °C/15 min	0.03 ± 0.00 a	5.41 ± 0.03 g	0.22 ± 0.01 d	0.22 ± 0.00 b	0.07 ± 0.00 bc	1.84 ± 0.01 b	0.02 ± 0.00 a	82.16 ± 0.05 h	9.76 ± 0.01 a	0.18 ± 0.01 a	0.16 ± 0.00 ab	0.06 ± 0.00 b	0.02 ± 0.00 c	0.04 ± 0.00 d	7.70	82.68	9.82
165 °C/15 min	0.03 ± 0.00 f	5.23 ± 0.04 d	0.22 ± 0.01 bc	0.22 ± 0.00 a	0.07 ± 0.00 cd	1.69 ± 0.01 a	0.05 ± 0.01 g	79.50 ± 0.04 b	12.69 ± 0.01 g	0.18 ± 0.01 c	0.17 ± 0.01 c	0.07 ± 0.01 f	0.03 ± 0.00 cd	0.04 ± 0.00 f	7.38	80.05	12.76
165 °C/30 min	0.03 ± 0.00 c	5.26 ± 0.01 e	0.19 ± 0.00 bc	0.19 ± 0.00 c	0.06 ± 0.00 ab	2.28 ± 0.01 d	0.03 ± 0.00 d	80.12 ± 0.01 d	11.53 ± 0.01 e	0.20 ± 0.01 b	0.15 ± 0.00 a	0.06 ± 0.00 b	0.02 ± 0.00 e	0.04 ± 0.01 b	7.99	80.58	11.59
185 °C/15 min	0.03 ± 0.00 b	5.51 ± 0.02 h	0.20 ± 0.00 b	0.20 ± 0.00 b	0.07 ± 0.00 f	1.97 ± 0.01 e	0.03 ± 0.00 e	77.89 ± 0.01 a	13.77 ± 0.02 h	0.20 ± 0.01 b	0.16 ± 0.00 ab	0.07 ± 0.00 d	0.02 ± 0.00 b	0.04 ± 0.00 e	7.94	78.39	13.84
200 °C/5 min	0.03 ± 0.00 b	4.98 ± 0.03 b	0.18 ± 0.00 b	0.18 ± 0.00 cd	0.07 ± 0.00 a	2.07 ± 0.01 f	0.01 ± 0.00 c	82.14 ± 0.09 g	10.00 ± 0.04 b	0.22 ± 0.01 ab	0.16 ± 0.00 ab	0.05 ± 0.01 a	0.02 ± 0.00 a	0.05 ± 0.00 a	7.49	82.61	10.05

[a] Mean ± SD, $n = 3$. Data followed by different letters within each column are significantly different according to Student's t tests at $P < 0.05$. SAT, saturated fatty acids; MUFA, monounsaturated fatty acids; PUFA, polyunsaturated fatty acids.

Table 3. Effect of Roasting on Triacylglycerol Composition[a]

	triacylglycerol (relative %)										
	LLL	OLL	PLL	OOL	POL	PPL	OOO	POO	PPO	SOO	PSO
raw	0.58 ± 0.02 d	4.26 ± 0.05 f	0.19 ± 0.01 d	19.96 ± 0.11 d	2.15 ± 0.05 c	0.02 ± 0.00 c	60.43 ± 0.31 b	9.82 ± 0.13 b	0.08 ± 0.01 bc	2.44 ± 0.19 ab	0.07 ± 0.01 a
125 °C/15 min	0.42 ± 0.02 bc	3.29 ± 0.12 d	0.13 ± 0.01 b	19.21 ± 0.08 c	1.72 ± 0.05 a	0.02 ± 0.00 ab	63.71 ± 0.22 d	9.20 ± 0.09 a	0.06 ± 0.00 a	2.18 ± 0.08 a	0.07 ± 0.00 a
125 °C/30 min	0.34 ± 0.02 ab	2.78 ± 0.07 c	0.13 ± 0.00 b	17.42 ± 0.34 b	1.87 ± 0.03 b	0.02 ± 0.00 bc	63.56 ± 0.10 d	10.46 ± 0.16 cd	0.09 ± 0.00 c	3.22 ± 0.01 cd	0.10 ± 0.01 c
145 °C/15 min	0.28 ± 0.01 a	2.19 ± 0.04 a	0.10 ± 0.00 a	16.49 ± 0.26 a	1.69 ± 0.03 a	0.02 ± 0.00 abc	65.63 ± 0.22 f	10.81 ± 0.05 d	0.09 ± 0.00 c	2.63 ± 0.06 b	0.07 ± 0.00 a
165 °C/15 min	0.45 ± 0.03 c	3.84 ± 0.06 e	0.15 ± 0.01 c	21.05 ± 0.24 e	2.15 ± 0.02 c	0.02 ± 0.00 bc	60.19 ± 0.39 b	9.77 ± 0.04 b	0.08 ± 0.01 bc	2.24 ± 0.03 a	0.06 ± 0.00 a
165 °C/30 min	0.35 ± 0.02 ab	3.06 ± 0.06 d	0.13 ± 0.00 b	19.07 ± 0.32 c	1.98 ± 0.03 c	0.02 ± 0.00 abc	61.21 ± 0.28 c	10.47 ± 0.10 cd	0.08 ± 0.00 bc	3.52 ± 0.04 d	0.10 ± 0.00 bc
185 °C/15 min	0.73 ± 0.05 e	4.61 ± 0.06 g	0.26 ± 0.00 e	20.76 ± 0.23 e	2.57 ± 0.02 d	0.02 ± 0.00 c	57.29 ± 0.30 a	10.50 ± 0.10 cd	0.11 ± 0.00 d	3.05 ± 0.29 c	0.09 ± 0.01 bc
200 °C/5 min	0.37 ± 0.04 b	2.54 ± 0.15 b	0.11 ± 0.01 ab	16.84 ± 0.14 ab	1.69 ± 0.04 a	0.02 ± 0.00 a	64.74 ± 0.16 e	10.21 ± 0.25 c	0.08 ± 0.00 b	3.32 ± 0.04 cd	0.09 ± 0.00 b

[a] Mean ± SD, $n = 3$. Data followed by different letters within each column are significantly different according to Student's t tests at $P < 0.05$. P, palmitoyl; S, stearoyl; O, oleoyl; L, linoleoyl.

Table 4. Effect of Roasting on Phytosterol Composition[a]

	phytosterol (mg/100 g of oil)									
	cholesterol	campesterol	stigmasterol	stigmastadienol	clerosterol	β-sitosterol	Δ^5-avenasterol	Δ^7-stigmastenol	Δ^7-avenasterol	total
raw	0.29 ± 0.02 bc	10.09 ± 0.16 d	1.85 ± 0.06 e	nd a	1.15 ± 0.08 a	155.96 ± 0.19 d	12.23 ± 0.28 cd	1.05 ± 0.03 a	0.98 ± 0.04 de	183.6
125 °C/15 min	0.29 ± 0.04 bc	9.57 ± 0.08 c	1.69 ± 0.03 cd	0.21 ± 0.02 b	1.13 ± 0.05 a	139.66 ± 1.06 c	9.94 ± 0.99 ab	1.62 ± 0.01 d	0.58 ± 0.05 c	164.7
125 °C/30 min	0.32 ± 0.01 c	8.97 ± 0.08 b	1.56 ± 0.05 ab	0.29 ± 0.04 c	1.13 ± 0.04 a	136.98 ± 0.9 b	9.89 ± 0.19 ab	1.02 ± 0.04 a	0.20 ± 0.02 a	160.4
145 °C/15 min	0.34 ± 0.01 c	8.76 ± 0.04 ab	1.78 ± 0.02 de	0.32 ± 0.01 c	1.25 ± 0.06 a	141.71 ± 0.59 c	12.43 ± 0.05 cd	1.25 ± 0.01 b	0.91 ± 0.01 d	168.7
165 °C/15 min	0.42 ± 0.04 d	8.96 ± 0.22 b	1.61 ± 0.01 abc	0.75 ± 0.02 d	1.33 ± 0.00 a	130.18 ± 1.00 a	10.85 ± 0.48 abc	1.93 ± 0.05 e	1.09 ± 0.08 e	157.1
165 °C/30 min	0.22 ± 0.02 ab	8.45 ± 0.19 a	1.66 ± 0.04 bc	0.87 ± 0.01 e	1.20 ± 0.06 a	134.89 ± 0.68 b	13.27 ± 1.31 d	1.64 ± 0.13 d	0.90 ± 0.1 d	163.1
185 °C/15 min	0.17 ± 0.02 a	9.06 ± 0.03 b	1.54 ± 0.01 a	0.19 ± 0.02 b	1.14 ± 0.08 a	134.92 ± 0.62 b	11.67 ± 0.35 bcd	1.17 ± 0.01 ab	0.38 ± 0.01 b	160.2
200 °C/5 min	0.16 ± 0.01 a	8.91 ± 0.08 b	1.67 ± 0.04 c	0.32 ± 0.02 c	1.21 ± 0.08 a	140.89 ± 0.68 c	9.23 ± 0.34 a	1.44 ± 0.09 c	0.27 ± 0.02 ab	164.1

[a] Mean ± SD, $n = 3$. Data followed by different letters within each column are significantly different according to Student's t tests at $P < 0.05$. nd, not detected.

Table 5. Effect of Roasting on Tocopherol and Tocotrienol Composition[a]

	tocopherol and tocotrienol (mg/kg of oil)							
	α-Toc	α-TTR	β-Toc	γ-Toc	β-TTR	γ-TTR	δ-Toc	total
raw	244.70 ± 5.25 d	1.88 ± 0.02 a	8.48 ± 0.07 e	9.72 ± 0.13 e	0.30 ± 0.01 cd	1.23 ± 0.02 c	0.99 ± 0.04 d	267.30
125 °C/15 min	236.22 ± 0.85 c	2.27 ± 0.09 b	8.07 ± 0.17 d	6.79 ± 0.17 c	0.35 ± 0.01 e	1.16 ± 0.03 bc	0.82 ± 0.02 c	255.68
125 °C/30 min	240.70 ± 0.29 cd	2.36 ± 0.03 b	9.11 ± 0.04 f	11.71 ± 0.02 f	0.31 ± 0.02 cd	1.21 ± 0.02 c	1.35 ± 0.01 f	266.75
145 °C/15 min	225.36 ± 0.19 b	2.77 ± 0.07 c	7.30 ± 0.08 b	6.99 ± 0.06 c	0.30 ± 0.02 cd	1.13 ± 0.02 b	0.72 ± 0.01 b	244.58
165 °C/15 min	230.15 ± 0.62 b	6.07 ± 0.02 g	7.81 ± 0.01 c	7.62 ± 0.05 d	0.28 ± 0.01 bc	2.47 ± 0.05 f	1.07 ± 0.01 e	255.47
165 °C/30 min	219.74 ± 0.30 a	4.41 ± 0.05 f	7.32 ± 0.02 b	5.40 ± 0.01 a	0.23 ± 0.01 a	1.31 ± 0.01 d	0.94 ± 0.01 d	239.35
185 °C/15 min	214.27 ± 0.17 a	3.53 ± 0.06 d	6.74 ± 0.02 a	5.49 ± 0.03 a	0.25 ± 0.01 ab	1.06 ± 0.01 a	0.60 ± 0.01 a	231.94
200 °C/5 min	228.31 ± 1.04 bc	4.09 ± 0.10 e	8.42 ± 0.09 e	6.59 ± 0.01 b	0.33 ± 0.01 de	2.10 ± 0.01 e	0.64 ± 0.01 a	250.49

[a] Mean ± SD, $n = 3$. Data followed by different letters within each column are significantly different according to Student's t tests at $P < 0.05$. Toc, tocopherol; TTR, tocotrienol.

TAG Composition. Changes in TAG composition during thermal processing have already been described for other matrices (*28*, *31*, *33*, *35*, *36*). **Table 3** shows the relative percentage of each triacylglycerol, for raw and roasted hazelnut samples. Eleven TAG were identified and quantified, which is in agreement with data already reported for TAG composition of raw hazelnuts (*37*−*39*). The results obtained are also consistent with the FA composition of raw and roasted hazelnuts presented in **Table 2**, because with the increase of temperature and roasting time, a decrease of TAG containing linoleic acid moieties and an increase of TAG containing oleic, palmitic, and stearic acids are observed. Again, roasting at 185 °C during 15 min was an exception, presenting a behavior similar to that observed for the fatty acid composition. The results are in good agreement with those already reported by Yoshida et al. when they studied the roasting effects on TAG content of peanuts, soybeans, sesame seeds, and sunflower seeds (*28*, *31*, *33*, *35*, *36*). These authors observed that, in general, as roasting time increased, losses of TAG species were more pronounced in those containing more than four double bonds, probably because the rate of FA breakdown was related to the increasing rate of oxidation with increasing unsaturation.

Phytosterol Composition. The roasting effects on the total and individual phytosterol contents (milligrams per 100 g of oil) are shown in **Table 4**. The values obtained are in agreement with those previously reported for raw hazelnuts (*20*). Changes in sterol contents can occur due to oxidation, hydrolysis, isomerization, and dehydration (*40*). In general, considering absolute values (milligrams per 100 g of oil), a slight decrease of individual and total phytosterols is observed in the roasted samples (8.0−14.4% of total phytosterols compared to raw hazelnuts). Δ^7-Stigmastenol was an exception, because higher values were found in the hazelnuts submitted to thermal processing. According to Piironen et al. (*40*) this increase may be due to the isomerization of β-sitosterol. Although the same authors also pointed out that severe physical refining can cause isomerization of Δ^5-avenasterol to Δ^7-avenasterol, higher levels of Δ^7-avenasterol were not observed under the used roasting conditions. Roasted hazelnuts were also distinguished from the raw sample by the presence of a characteristic peak which, on the basis of literature data (*37*, *41*), was tentatively identified as $\Delta^{5,23}$-stigmastadienol. Stigmastadienes are sterol dehydration products and are known to occur when foods are submitted to high temperatures (*42*). The presence of stigmastadienes occurs also in commercially refined vegetable oils, where they can be present in levels ranging from 1 to 100 mg/kg.

Considering that all roasting procedures showed total phytosterol losses below 15%, the results presented in **Table 4** are in agreement with those reported by Crews et al. (*30*), who reported the absence of significant effects of roasting on the desmethylsterol composition of hazelnuts.

Considering the changes in each compound relative level, it can be observed that $\Delta^{5,23}$-stigmastadienol, Δ^5-avenasterol, and Δ^7-stigmastenol were the most affected compounds but, in general, only slight differences were observed among raw and roasted hazelnuts. These results are different from those reported by Mohamed et al. (*26*), but in accordance with those reported by Yoshida et al. (*28*, *33*), who found no statistically significant decreases of steryl esters occurring during microwave roasting of peanuts and that significant loss was found to occur only after 30 min of microwave roasting of sunflower seeds.

Sterols with an ethylidene group in the side chain (Δ^5- and Δ^7-avenasterols) have been described as possessing antipolymerization properties (*43*), thus protecting the oils from oxidation during thermal processing. Although some minor decreases of both these sterols occurred with roasting, their presence may still be beneficial.

Tocopherol Composition. The individual and total tocopherol and tocotrienol contents for raw and roasted hazelnuts are shown in **Table 5**. Seven compounds were detected and quantified (α-, β-, γ-, and δ-tocopherols and α-, β-, and γ-tocotrienols). In all samples α-tocopherol was the predominant compound, followed by β- and γ-tocopherols. Considering total contents of vitamin E, losses were below 10% for all roasting treatments, the higher loss (10%) being achieved when hazelnuts were roasted at 185 °C during 15 min. This finding is in conformity with the study of Yoshida et al. (*28*), who reported that after 20 min of roasting, the amount of tocopherol homologues was still >85% of the original levels in peanuts.

Owing to the antioxidant properties and several possible benefits already ascribed to vitamin E homologues, these compounds are thought to present positive nutritional effects in humans. For this reason, the study of their individual contents is of great importance. In this study, with increasing roasting times and temperatures, the several vitamers presented different behaviors. α-Tocopherol content showed a slight decrease, achieving the lowest content when hazelnuts were roasted at 185 °C during 15 min (9.2% less than control). Some studies have pointed out that at high temperature α-tocopherol is the least stable tocopherol homologue (*44*, *45*), whereas other studies have demonstrated that this vitamer can be rather stable to heat conditions (*46*). In our study, considering the results obtained for all of the tocopherol homologues, α-tocopherol seemed to be the one less affected by high temperatures. With respect to the relative stability of the other homologues, δ-tocopherol has been described as the most stable, whereas β- and γ-tocopherols were found to present intermediate degradation rates (*44*, *45*). The results herein reported are not necessarily

in complete agreement with literature data, because γ-tocopherol was the vitamer that exhibited the higher loss and δ-tocopherol the second one. Moreover, the degradation rates of β- and γ-tocopherols were very different from each other. In general, γ-tocopherol seemed to be more affected by thermal processing because it presented higher relative losses compared to β-tocopherol; compared to raw hazelnut values, this last vitamer presented the highest loss when roasting occurred at 185 °C during 15 min (17.6%), whereas γ-tocopherol presented the highest loss at 165 °C during 30 min (42.5%).

The absolute amounts of α-tocotrienol seemed to increase with temperature and roasting periods. This was not a new finding as other authors have also reported this fact for other homologues (*27*, *46*, *47*). These authors suggested that the compounds for which content increased were bound to membrane proteins or linked to other compounds, such as phospholipids, and that heat treatment could have caused the breakdown of these bonds and, consequently, a higher yield of extraction. In a previous work carried out with hazelnuts in our laboratory (*25*), the extraction procedure herein used (extraction with *n*-hexane, at ambient temperature) was compared with a procedure involving an alkaline hydrolysis. In that work it was found that alkaline hydrolysis allowed the extraction of higher amounts of γ-tocopherol (> 7%) and γ-tocotrienol (> 27%). In that same work, an extraction using a Soxhlet apparatus was also tested, causing a 42% higher yield of α-tocotrienol. These findings corroborate the idea that heat and hydrolysis are involved in the higher extractability of these compounds. Nevertheless, the higher extractability does not justify the 3 times higher α-tocotrienol content of the 165 °C/15 min roasting when compared to the raw values. It should be noted that for roasts above 145 °C, the analysis of DAD spectra allowed us to conclude that α-tocotrienol coeluted with another compound, probably a tocochromanol degradation product. This unidentified compound presented an absorption maxima at λ 270 nm, instead of the λ 293 nm expected for α-tocotrienol. As the compounds were coeluting, they were both quantified as α-tocotrienol, explaining the abnormally high values obtained.

The γ-tocotrienol contents were also higher for some of the roasted samples, probably because of the higher extractability of the vitamer caused by the heat treatment. β-Tocotrienol was present in low amounts in raw and roasted hazelnuts, and its content was more or less identical in all samples. As for γ-tocopherol it seems that two phenomena occurred during the assay: on the one hand, a considerable decrease of its level when heat is applied can be seen (especially noted at 165°C/30 min and 185°C/15 min, when decreases reach 42.5%); on the other hand, when some treatments with increasing temperatures and the same time of exposure are compared (125, 145, and 165 °C, all during 15 min), there is a rise of its levels, suggesting the effect of improved extractability.

In conclusion, from the results herein presented it seems that, as far as the compounds studied are concerned, the nutritional value of hazelnuts is not substantially affected by roasting, even at temperatures higher than those used by the industry. Although the extent of losses presented some tendency to increase with higher temperatures and roasting periods, these experiments revealed that roasting caused only a modest decrease in the total levels of the beneficial phytosterols (maximum of 14.4%) and vitamin E (maximum of 10.0%) compounds. A negligible increase of the potentially harmful trans fatty acids also occurred. Although some minor changes occurred in the triacylglycerol and fatty acid compositions, the corresponding profiles basically remained identical to that of raw hazelnuts. Considering each group of compounds separately, the relative proportions of the individual fatty acids, triacylglycerols, sterols, tocopherols, and tocotrienols were essentially not altered in the roasted samples.

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