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Effects of Different Roasting Conditions on Physical-Chemical Properties of Polish Hazelnuts (*Corylus avellana* L. var. *Kataloński)*

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Abstract

The influence of different roasting conditions on the physical-chemical (water activity, moisture, colour, volatile compounds, tocopherols, phenolic content) properties of Polish hazelnuts (cv. *Kataloński*) was determined. Nuts were roasted at specific temperature/time conditions: 130 °C/40 min, 130 °C/50 min, 130 °C/60 min, 160 °C/20 min, 160 °C/25 min, 160 °C/30 min. Hazelnuts roasted at 160 °C showed a darker colour and a lower water activity and moisture than samples roasted at 130 °C. Compared to raw hazelnuts, the phenolic content increased in all roasted samples, although with a more concentration in nuts roasted at 160 °C (2998.84 mg/100 g, 3429.52 mg/100g and 2927.81 mg/100 g after 20, 25 and 30 min respectively). The different roasting conditions led to several aroma modifications, in fact in raw hazelnuts were identified just only 22 compounds, whereas in samples roasted at 130 °C and 160 °C were found 79 and 102 volatile compounds, respectively.

Keywords: hazelnut, roasting, tocopherols, phenolic compounds, volatile compounds

1. Introduction

Hazelnut (*Corylus avellana* L.) belongs to the family of *Betulaceae* and is one of the most popular nuts worldwide; it is produced especially in the coasts of Black Sea region of Turkey, in southern Europe (Italy, Spain, Portugal and France) and in some areas of the United States (Oregon and Washington). Furthermore hazelnuts are grown in New Zealand, China, Azerbaijan, Chile, Iran, Georgia, Kirgizstan, Poland and Croatia (Pelvan, Alasalvar, & Uzman, 2012; Ciemniewska-Żytkiewicz, Verardo, Pasini, Bryś, Koczoń & Caboni, 2015b).

The biochemical composition of hazelnuts has been extensively studied because of their health promoting properties and their good source of energy due to a fat content of about 60% (Ciemniewska-Żytkiewicz, et al., 2015b). Hazelnuts provide also essential minerals (Ca, Mg, P, K), vitamins E and B complex, fibres and amino acids. Moreover, several studies have shown that hazelnuts are rich in some antioxidant compounds, such as tocopherols and polyphenols, which exhibit a beneficial effect on human health, reducing oxidative stress and risk of cancer, stroke, inflammation, and other neurodegenerative diseases (Yurttas, Schafer & Warthesen, 2000; Kornsteiner, Wagner & Elmadfa, 2006; Shahidi, Alasalvar & Liyana-Pathirana, 2007). Besides, phenolic compounds contribute greatly to some hazelnuts organoleptic properties, such as astringency and bitterness (Cristofori, Ferramondo, Bertazza & Bignami, 2008).

Roasting process is carried out to remove the pellicles of kernels, inactivate enzymes, destroy microorganisms and reduce water activity (Özdemir, Seyan, Bakan, İlter, Özay & Devres, 2001); moreover, roasting is used to improve the colour, the crispy texture and the flavour of the product (Burdack-Freitag & Schieberle, 2010). The thermal treatment applied during roasting processes leads to physical changes such as dehydration (Amaral, Casal, Seabra & Oliveira, 2006), colour modifications (Alamprese, Ratti & Rossi, 2009), biochemical changes including lipid structure modification (Amaral et al., 2006) and Maillard reactions that give

rise to pyrazines compounds associated with the development of typical roasted flavour (Saklar, Katnas & Ungan, 2001).

The roasting conditions generally used for hazelnuts are in a range from 100 to 160 °C for 10 to 60 minutes (Donno et al., 2013). Ciemniewska-Żytkiewicz, Bryś, Bryś, Sujka & Koczoń (2014) roasted hazelnut *Kataloński* variety at three temperatures (100, 130, 160°C), of which 130 and 160 °C were reported as the most suitable for hazelnut sample final characteristics. Roasted hazelnuts are used in food production such as chocolate spreads, ice creams, cereal bars, cookies, etc. (Cucu, Platteau, Taverniers, Devreese, de Loose & de Meulenaer, 2011).

Different authors studied the influence of roasting conditions on physical-chemical properties of hazelnuts. Ciemniewska-Żytkiewicz et al. (2014) showed a decrease of moisture content according to the temperature/time conditions and a change of hazelnuts' colour with a decrease of L^* and a^* values compared to raw samples. Schmitzer, Slatnar, Veberic, Stampar & Solar (2011) and Pelvan et al. (2012) have observed a loss in phenol content of about 66.3% in roasted hazelnuts in respect to raw ones, due to the removal of the skin which contains the majority of phenols. Some authors investigated also the trend of tocopherols during roasting: Schlörmann et al. (2015) showed a decrease of α and β-tocopherols after roasting treatment of about 34% and 40%, respectively, whereas Amaral et al. (2006) found only a reduction of 9% of α-tocopherol content at roasting conditions of 185 °C/15 min, as compared to raw hazelnuts. Finally, Alasalvar, Shahidi & Cadwallader (2003a) have compared the volatile compositions of raw and roasted hazelnuts (165 °C/25 min). After roasting, hazelnut volatile profile was more concentrated and rich in new other compounds, not present in the raw samples.

During roasting a lot of volatile compounds, belonging to ketones, aldehydes, pyrazines, alcohols, aromatic hydrocarbons, furans, pyrroles, terpenes and acid classes are released from hazelnuts; among these compounds, the 5-methyl-(*E*)-2-hepten-4-one (filbertone) has been

reported as primary odorant (nutty-roasty and hazelnut-like) of roasted hazelnuts (Langourieux, Perren & Escher, 2000; Alasalvar et al., 2003a).

Studies regarding the effects of roasting on *Kataloński* hazelnut variety are limited in literature (Ciemniewska-Zytkiewicz et al., 2014; Ciemniewska-Zytkiewicz, Bryś, Sujka & Koczoń, 2015a; Ciemniewska-Zytkiewicz et al., 2015b); therefore this research was conducted in order to evaluate the influence of different roasting conditions on some physical and chemical characteristics of this Polish variety. Obtained results were compared and related to available literature data.

2. Materials and Methods

2.1. Chemicals

All the solvents and reagents for phenolic compounds and lipid extraction were from Sigma Aldrich (Saint Louis, MO, USA). Folin Ciocalteu's reagent was purchased from MercK (Darmstadt, Germany) and Na_2CO_3 for the determination of total phenolic content was from BDH AnalaR® (Poole, England). All the solvents for the determination of tocopherols were supplied by VWR Prolabo Chemicals (Dublin, Ireland).

2.2. Samples

Kataloński variety hazelnuts (*Corylus avellana* L.) were obtained from an orchard located in the south of Poland (Jankowice, Pszczyna 50°0′ 5″ N 18°59′ 18″ E) in 2013. Hazelnuts were collected at complete maturity, sun-dried for 3 days at 20-25 °C and stored with shell at 4 °C until the analysis.

Hazelnuts were manually cracked and shelled with a nutcracker before roasting. The fibrous skin, particularly distinctive for *Kataloński* variety, was removed by hands. Before the analyses the hazelnut samples were ground with a blender (Moulinex, France).

2.3. Roasting of hazelnuts

Approximately 50 g of shelled hazelnuts were roasted in a lab-scale ventilated oven (Vismara, Italy) at different time and temperature conditions: 130 °C (Low Temperature, LT) for 40 (1), 50 (2) and 60 (3) minutes, and 160 °C (High Temperature, HT) for 20 (4), 25 (5) and 30 (6) minutes. Each roasting protocol was carried out three times.

For each roasting cycle, temperature data were recorded every 15 s during the experiment using a digital multimeter mod. SCC-TC02 (National Instruments, Assago, MI, Italy) coupled with thermocouples and a personal computer. During all roasting tests, three thermocouples were inserted inside three hazelnuts by the help of a tip needle, in order to measure the temperature profile in the kernel core during the heating process. One thermocouple was also positioned inside the oven in a central point in which the oven temperature represented the average value according to results of preliminary experiments.

2.4. Moisture and water activity determination

Water activity (a_w) was measured at 20 ± 2 °C on 3 replicates of grounded hazelnuts for each sample with a dew point hygrometer Aqualab® series 3 TE (Decagon Devices Inc., Pullman, WA., U.S.A.).

Water content (%) was evaluated on ground hazelnut samples in an oven at 105 °C until constant weight was reached. For each sample, 3 replicates of 3 g weighted were dried (AOAC, Official Methods of Analysis, ed.by Horwitz E. AOAC,Washington, DC (1980).

151

152 *2.5. Colorimetric analysis*

153 The colour of chopped hazelnuts was measured with a colour spectrophotometer mod.
154 Colorflex (Hunterlab, USA) equipped with a measuring head (diameter 127 mm). Colour was
155 measured using the CIE L*a*b* scale and illuminant D65. The instrument was calibrated with
156 a white tile (L* = 98.03, a* = -0.23, b* = 2.05) and the calibration was also validated with
157 green standard tile (L* = 53.14, a* = -26.23, b* = 12.01) before the measurements. The
158 hazelnut's colour was described in terms of luminosity (L*) and red index (a*). The results
159 are the mean of 10 measurements for each sample.
160 Browning index (BI) was also calculated based on CIE L*a*b* coordinates, using the
161 following expression (Mohapatra et al., 2010):

162 $$BI = 100 \times \left(\frac{X - 0.31}{0.17} \right),$$

163 where,

164 $$X = \frac{(a* + 1.75L)}{(5.645L + a* - 3.012b*)},$$

165

166 *2.6. Extraction of phenolic compounds*

167 To collect the phenolic fractions, the extraction protocol of Ciemniewska-Żytkiewicz et al.
168 (2015b) was used. Approximately 3 g of ground hazelnut kernels were defatted by *n*-hexane
169 and then extracted in an ultrasonic bath using 30 mL of ethanol/water solution (4/1 v/v) at 40
170 °C for 15 minutes. After centrifugation at 3500 rpm for 15 minutes, the supernatant was
171 collected and the residue was re-extracted under the same conditions. Supernatants were
172 pooled, evaporated at 35 °C with a vacuum evaporator, and reconstituted with 2 mL of

methanol-water (1/1 v/v). Each extraction was carried out two times for each set of roasting (*n* = 6 for roasting condition) and the extracts were stored at -18 °C until use.

2.7. Determination of total phenolic content

The total phenolic content (TPC) of the extracts was assessed by means of the Folin-Ciocalteu method (Singleton & Rossi, 1965). Briefly, 100 µL of each extract was shaken with 500 µL Folin–Ciocalteu reagent and 6 mL of distilled water. Two millilitres of 15% Na_2CO_3 was added and the mixture was shaken once again for 30 seconds. Finally, the solution was brought up to 10 mL by adding distilled water. After 2 h, the absorbance at 750 nm was measured using glass cuvettes. The phenolic content was calculated on the basis of the gallic acid calibration curve (from 25 to 1000 µg/mL). Absorptions were measured in 2 replicates for each extract (*n* = 12 for roasting condition) and the results were expressed as mg/100g of hazelnuts d.w.

2.8. Extraction of lipid fraction

According to Verardo, Bendini, Cerretani, Malaguti, Cozzolino. & Caboni (2009), the lipid fraction was extracted from ground hazelnuts (3 g) with diethyl ether in a Soxtec apparatus (System HT 1046 Service Unit Tecator, Apeldoorn, The Netherlands). The oil was taken up with *n*-hexane/isopropanol (4/1 v/v) solution and stored at -18 °C until use. Each extraction was carried out two times for each set of roasting (*n* = 6 for all roasting conditions).

2.9. Tocopherols analysis

For the tocopherols determination, approximately 0.05 g of fat was dissolved in 0.5 mL of *n*-hexane. The solutions were filtered through a 0.45 µm nylon filter. The tocopherols were determined by HPLC (Agilent 1200 series, Palo Alto, CA, USA) equipped with a fluorimeter detector (Agilent, Palo Alto, CA, USA). The excitation wavelength was 290 nm and the emission one was 325 nm. The column used was a Luna Hilic Phenomenex column (250 mm x 4.6 mm i.d., 5 µm particle size) in isocratic conditions according to Gómez-Caravaca, Verardo & Caboni (2010). The calibration curve was constructed with α-tocopherol standard solution (from 1 to 100 µg/mL) and it was used for quantification.

2.10. SPME-GC-MS analysis

Headspace volatiles from each hazelnut sample roasted at different roasting conditions were analysed by headspace solid phase microextraction-gas chromatography/mass spectrometry (HS-SPME-GC/MS), using a GC-MS–QP2010 Plus (Shimadzu, Tokyo, Japan) equipped with an AOC 5000 autosampler (Shimadzu, Tokyo, Japan).

About 3 g of ground hazelnut were weighed into a 10 mL amber vial, crimped with aluminium caps equipped with a septum. The samples were equilibration at 40 °C for 30 minutes; a 2 cm x 0.11 µm (i.d.), 50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/Carboxen/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was then inserted through the septum into the vial at 40 °C for another 10 minutes; vial penetration depth was 20 mm. Afterwards, the SPME fiber was desorbed at 240 °C for 7 minutes in the split mode. An Rtx-Wax fused-silica capillary column (30 m × 0.25 mm i.d. × 1.0 µm f.t.) (Phenomenex, Torrance, CA, USA) was used for the chromatographic separation. The oven was programmed from 40 °C (kept for 10 minutes) to 200 °C at 3 °C/min and maintain it for 3 minutes, then increased from 200 °C to 240 °C at 10 °C/min and kept at the final temperature

for 5 minutes. The injector, transfer line and the ion source temperatures were set at 240 °C, 240 °C and 200 °C respectively. Helium was used as the carrier gas at an inlet pressure, constant flow rate of 1.5 mL/min; the split ratio was 1:10.

The filament emission current was 70 eV. A mass range from *m/z* 30 to 250 was scanned from 3.5 to 70 minutes. The acquisition was carried out in Total Ion Current (TIC) mode, using the GCMS solution software, version 2.50 SU1 (Shimadzu, Tokyo, Japan). Each extraction was carried out two times for each set of roasting (n = 6 for roasting condition).

Identification of volatile compounds was performed by comparing their mass spectra with those reported in literature and the NIST Mass Spectral Database (NIST 08, National Institute of Standards and Technology, Gaithersburg, MD, USA).

2.11. Statistical Analysis

Relative standard deviation was obtained, where appropriate, for all data collected. One-way analysis of variance, ANOVA (Tukey's honest significant difference multiple comparison) and Principal Component Analysis were evaluated using Statistica 8 software (2006, StatSoft, Tulsa, OK, USA). *p*-values lower than 0.05 were considered statistically significant.

3. Results and discussion

The thermal profiles of oven and hazelnuts samples obtained during roasting processing at 130 °C (LT) and 160 °C (HT) for different times are respectively reported in Figures 1A and 1B. As show in Figure 1A the temperature inside roasted hazelnuts reached a plateau around 130 °C between 25 to 30 minutes of treatment, whereas roasted hazelnuts at 160 °C showed an increase of temperature inside the product more abruptly, reaching 130-135 °C during the first 8-10 minutes (Figure 1B).

After both roasting treatments it was difficult to remove the fibrous skin that is present between shell and thin skin, particularly distinctive for *Kataloński* variety that remained adhered to the hazelnuts.

3.1. Moisture, water activity and colour

Roasting levels reached by hazelnut samples were assessed on the basis of roasting parameters, such as moisture, water activity and colour (L*, a* and BI), that represent the most important physical changes that occur in the product during the thermal process. In Table 1 moisture, water activity and colour data of all hazelnut samples before and after roasting are reported.

The moisture of raw kernels was 4.86% on average; this result agreed with those reported in literature (Amaral et al. 2006; Saklar et al., 2001) for different hazelnut varieties. As expected, moisture content decreased significantly in the samples proportionally to the intensity of applied process in terms of temperature/time conditions. The sample roasted for 30 minutes at 160 °C underwent the highest water loss.

The water activities of raw samples were significantly higher as compared to roasted kernels, among which statistically important differences were not found for the LT samples, whereas the HT roasted samples showed a significant a_w decrease after 30 minutes of roasting.

Colour is an important indicator of roasting level of hazelnuts. In these kind of products it is described that the formation of browning substances is a result of Maillard type nonenzymatic reactions between reducing sugars and free amino acids or amides (Moss & Otten 1989; Cammarn, Lange & Beckett 1990; Donno et al., 2013). Donno and co-workers (2013) reported that the *b**-value is not so suitable for monitoring hazelnut roasting level, since it is not fully affected by roasting conditions. Conversely, the *L**-value (relative lightness of product) is an ideal for monitoring colour development in roasted hazelnuts, because this

colour attribute is analogous to the colour observation made by the operator (Moss & Otten 1989; Ozdemir & Devres 2000). In this work browning index (BI) was also calculated, in order to more precisely evaluate the overall colour changes in hazelnut samples during roasting.

Herein, in hazelnut samples *L**-values decreased and *a** and BI values increased with the increase of roasting temperature and time (Table 1). These data prove the colour development from pale to brown during the hazelnut roasting process and it is in agreement with the trend reported by other authors (Donno et al., 2013; Saklar et al., 2001). However, the fibrous skin that partially remained on the fruit contributed to darker colour of hazelnut samples if compared with data obtained in previous works (Saklar et al., 2001; Donno et al., 2013; Ciemniewska-Żytkiewicz et al., 2014).

3.2. Total phenolic content (TPC) and tocopherols composition

In order to evaluate the evolution of principal antioxidant compounds, TPC and tocopherols changes during roasting were monitored.

As shown in Table 2 a large variation in TPC among raw and roasted hazelnut samples was observed. Raw hazelnuts had a total phenolic content of 1245.27 mg/100g d.w.; the highest values were recorded in LT1 (2017.27 mg/100g d.w.) and LT2 samples (2218.25 mg/100g d.w.), however changes of TPC within samples roasted at 130 ºC were not significant. Compared to the raw sample, the TPC increased also in HT samples; the highest value was reached for middle operation temperature (HT5) (3429.52 mg/100 g d.w); as observed for roasting at 130°C, HT6 demonstrated reduced phenolic content by 14.6% (2927.81 mg/100g d.w.) when compared to HT5.

HT samples demonstrated a TPC approximately 49.3% higher than LT ones; this could be explained by an easier extraction of phenolic substances linked to the matrix, due to a greater matrix destructuration when a higher temperature is applied. Nevertheless, the trends of the total phenolic content in roasted hazelnuts at 130 °C and 160 °C were similar, with an increase in the two sample roasted for the central times and a decrease in LT3 (130 °C/60min) and HT6 (160 °C/30min) ones.

Results obtained within this study were in disagreement with those reported by Pelvan et al. (2012) and Schmitzer et al. (2011) where authors obtained a lower TPC value in hazelnuts after roasting treatment; indeed Schmitzer and co-workers observed that the skin removal and applied roasting conditions (15 min at 140° C) affected the total phenolic content negatively. Several authors (Shahidi et al., 2007; Alasalvar, Karamać, Kosińska, Rybarczyk, Shahidi & Amarowicz, 2009b; Locatelli, Travaglia, Coïsson, Martelli, Stévigny & Arlorio, 2010) affirmed that the skin has a key role in the determination of total phenolic content containing the majority of them. The presence of skin in a part of *Kataloński* variety hazelnuts roasted in these trials could be contributed to significantly affecting the obtained TPC levels.

The individual tocopherols identified are shown in Table 2; the results were in agreement with literature results (Ciemniewska-Żytkiewicz, et al., 2015b, Alasalvar et al., 2003b; Amaral et al., 2006); in all samples α-tocopherol was the predominant compound followed by γ-tocopherol and β-tocopherol.

In raw samples, α-tocopherol content was 73.90 mg/100g of oil, β-tocopherol was 5.24 mg/100g and γ-tocopherol was 2.01 mg/100g; however, roasting conditions significantly affected the tocopherols contents ($p<0.05$), as compared to raw hazelnuts.

Amaral et al. (2006) declared that α-tocopherol is the less stable at high temperature, among the tocopherols identified; instead Seybold, Fröhlich, Bitsch, Otto & Böhm (2004) have

shown that this compound can be stable during heat treatment. Results obtained herein were much higher than those obtained in other studies (Ciemniewska-Żytkiewicz et al. 2014; Amaral et al., 2006; Schlörmann et al., 2015) but it could be explained by different harvest year as compared to Ciemniewska-Żytkiewicz et al. (2014) and protective role of additional skin wrapped around the kernels. Also in this case the presence of the skin in part of samples could give a protection to samples preventing a possible tocopherols thermal degradation.

3.3. Volatile compounds

SPME-GC-MS analysis identified 22 compounds in raw hazelnuts, 79 in hazelnuts roasted at 130°C and 102 in those roasted at 160°C. Compounds identified were ketones, aldehydes, pyrazines, furans, aromatic hydrocarbons, alcohols, terpenes and acids. In particular, pyrazines, terpenes, pyrroles, furans and acids were identified only in roasted hazelnuts. As reported by Alasalvar et al. (2003a) the compounds more responsible of roasted hazelnuts aroma are ketones, aldehydes, pyrazines and furans.

Among the 79 compounds detected in LT samples, 21 were identified as ketones, 10 as aldehydes, 11 as pyrazines and 4 as furans; whereas in HT samples 27 compounds were identified as ketones, 11 as aldehydes, 13 as pyrazines and 5 as furans.

Ketones. Because of their low threshold of perception, ketones play the most important role in the flavor profile of hazelnuts. Table 3 shows all the ketone compounds identified in the different samples with their respective areas. The ketones mainly present in hazelnut were 3-methyl-2-pentanone, 2,3-pentanedione, 3-penten-2-one, 5-methyl-(E)-2-hepten-4-one, 3,5-dimethyl-4-heptanone and 4-hexen-3-one.

Among these, 5-methyl-(E)-2-hepten-4-one (filbertone) has been reported by several authors (Alasalvar et al., 2003a; Burdack-Freitag & Schieberle, 2010; Nicolotti, Cordero, Bicchi,

Rubiolo, Sgorbini & Liberto, 2013) to be the compound that contributes to the typical nutty-roasty and hazelnut-like aroma of this nut. In samples roasted at both tested conditions, its concentration had no linear trend; in fact decreased from LT1 to LT2 samples and from HT5 to HT6 samples, moreover increased from LT2 to LT3 samples and HT4 and HT5. The sample that had the highest concentration of filbertone was HT5. In different studies (Burdack-Freitag & Schieberle, 2012; Kiefl & Schieberle, 2013), only the concentration of filbertone increased after roasting. These differences show that the concentration of this compound can depend not only on conditions of roasting process but also on variety and the fibrous skin presence. Moreover, other ketones play an important role in hazelnut aroma, like 3-penten-2-one that is responsible for fruity odour (Langourieux, Perren & Escher, 2000) and 2,3-pentanedione, a sugar degradation product responsible for sweet odour (Ho & Carlin, 1989). Concentration of both compounds increased in HT samples, in particular HT5 had the highest contents of them, whereby fruity and sweet odours increased after roasting process in 160 ºC significantly, which is expected by consumers.

Aldehydes. A total of 11 aldehydes were found in both LT and HT samples (Table 3). According to Alasalvar et al. (2003a) the predominant aldehydes were 2-methylpropanal, 2-methylbutanal and 3-methylbutanal. According to Burdack-Freitag & Schieberle (2012), the concentration of these compounds, in particular of 3-methylbutanal, increased after roasting causing simultaneously the increase of fruity, malty and chocolate-like odors (Alasalvar et al., 2003; Burdack-Freitag & Schieberle, 2010). As reported for ketones, the HT5 sample was the richest in these three aldehydes among all samples tested. Other aldehydes, 2-methyl-(E)-2-butenal and nonanal were identified only in LT samples, however the (E)-2-hexenal and 5-methyl-2-furancarboxaldehyde were present only in HT samples.

Pyrazines. A total of 11 pyrazines were detected in LT samples, whereas 14 compounds in HT samples, in particular, 2,3-dimethyl-5-ethylpyrazine, 2-ethenylpyridine and N-acetyl-4(H)-pyridine were present only in HT samples. These compounds, originated by Maillard reaction from free amino acids and monosaccharides, contribute to nutty and roasty aroma (Kiefl, Pollner & Schieberle, 2013a). According to Alasalvar et al. (2003a) the most abundant compounds were 2,5-dimethylpyrazine and methylpyrazine; moreover, herein, 2,6-dimethylpyrazine was one of major compound present in roasted hazelnut as well. The concentration of all pyrazines increased with increasing roasting time, as reported in Table 3. The highest content of these three pyrazines was detected in HT6 followed by HT5 sample. Kiefl et al. (2013a) in their study observed the same trend, where pyrazines were rapidly formed during roasting and they were correlated with aroma of hazelnuts and, in general, of processed food.

Furans. As reported for pyrazines, also furans are originated from Maillard reaction (Alasalvar et al., 2003a). Şenyuva & Gökmen (2007) reported that the formation of furans in hazelnuts during heat treatment increased at temperature exceeding 120 °C. In this study, furans were absent in raw hazelnuts and a total of 4 and 5 furans were detected in LT and HT samples, respectively. 2-ethyl-5-methylfuran was present only in LT samples, however, 2-methylfuran and 2,3-dihydro-4-methylfuran were identified only in HT samples. 2,5-dimethylfuran was the most abundant compound in LT3 and HT6, in agreement with results obtained by Alasalvar et al. (2003a), followed by 2,3,5-trimethylfuran that was higher in HT5 samples.

Figure 2A shows a PCA plot obtained with the areas of single volatile compounds, with 60.73% of the variation accounted for PC1 and 19.08% accounted for PC2. The figure shows a good separation between hazelnut samples, in particular raw hazelnuts (R) has been well

separated from LT and HT ones. Raw sample, that had positive scores according to both PC1 and PC2, was discriminated from the other samples for the nonanal. PC1 discriminated HT samples, that exhibited negative scores and LT samples that had positive scores. 2-pentanone, 4-ethyl-3-hexanone, 3-hepten-2-one, 2-hydroxy-2,4-dimethyl-3-pentanone, hexenal, 2-methyl-(E)-2-butenal, 2,5-diethyl-pyrazine, 2-ethyl-5-methyl-furan were responsible for PC1 discrimination of the samples. For PC2, only R and HT6 samples exhibited positive scores, all the other samples had negative scores. Some compounds responsible for this discrimination were 2,5-dimethyl-4-hydroxy-3(2H)-furanone, dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone, butyrolactone, 2-ethyl-6-methyl-pyrazine, N-acetyl-4(H)-pyridine, methyl-pyrazine, ethyl-pyrazine, 3-ethyl-2,-dymethil-pyrazine and 2,3-dihydro-4-methyl-furan.

As show in Figure 2B, PC3 explaining 9.68% variation demonstrated a good discrimination among R, LT and HT samples. For PC1 discrimination of R, LT and HT samples was the same of the case show in Figure 2A. For PC3 all the LT samples and HT6 exhibited positive scores and only R, HT4 and HT5 had negative scores. Some compounds responsible for PC3 discrimination were: 3-methyl-4-hexen-2-one, 1-(2-furanyl)-ethanone, 2,3-butanedione, 3-hydroxy-2-butanone, 2,3-pentanedione, 1-hydroxy-2-butanone, 2-methyl-propanal, trimethyl-pirazine, 2-ethyl-5-methyl-pyrazine, 2,5-dimethyl-pyrazine, 2,3-dihydro-4-methyl-furan, N-acetyl-4(H)-pyridine, 2,6-dimethyl-pyrazine and pyridine.

4. Conclusions

This study confirms that time and temperature used for hazelnuts roasting treatment can deeply influence their final quality. Obtained results show that the low temperature and short roasting time are not always the most appropriate way to obtain the best product, in terms of total phenolic content, tocopherols and volatile profile. In fact the highest concentration of volatile compounds, that are directly related also to hazelnut aroma, were obtained when high

420 temperature and long times had been used. Moreover, the presence in *Kataloński* hazelnuts of
421 a fibrous skin, difficult to remove after roasting process, significantly affected the roasting
422 process itself and the final quality characteristics of the product. The fibrous skin, in
423 particular, positively influenced the total phenolic content, which was higher if compared to
424 literature data obtained from different hazelnut varieties. This can improve health benefits of
425 roasted hazelnuts product, but could be also less acceptable by producers for further
426 processing and by consumers themselves.
427

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Captions of figures

Figure 1. Thermal profiles of oven and hazelnuts samples roasted at 130 °C (A) and at 160 ºC (B). Temperature data were recorded every 15 s during the experiment with thermocouples inserted inside three hazelnuts and one positioned inside the oven.

Figure 2. PCA score plots (PC1 x PC2, figure 2A) (PC1 x PC2, figure 2B) of volatile compounds of raw and roasted hazelnuts samples

Table 1

Moisture (%), a_w and colour (L*, a* and Browning Index - BI) of raw and roasted hazelnut samples obtained at different roasting conditions. Different letters in the same column showed significant differences ($p<0.05$).

Sample	Roasting conditions	Moisture (%)	a_w	L*	a*	BI
Raw		4.86 ± 0.19a	0.506 ± 0.002a	64.40 ± 0.27a	4.52 ± 0.10e	42.02 ± 0.34e
LT 1	130°/40min	1.48 ± 0.07b	0.192 ± 0.004bc	56.14 ± 0.49b	9.06 ± 0.32d	78.02 ± 1.91d
LT 2	130°/50min	1.12 ± 0.12c	0.176 ± 0.016bcd	55.64 ± 1.80b	9.54 ± 0.54d	79.72 ± 5.56d
LT 3	130°/60min	0.78 ± 0.20d	0.175 ± 0.036bcd	50.59 ± 2.14c	11.05 ± 0.38c	95.31 ± 4.41c
HT 4	160°/20min	1.33 ± 0.19bc	0.214 ± 0.039b	47.42 ± 0.78d	12.61 ± 0.50b	111.01 ± 7.10b
HT 5	160°/25min	0.83 ± 0.08d	0.164 ± 0.014cd	43.17 ± 1.05e	13.40 ± 0.36a	120.39 ± 8.45a
HT 6	160°/30min	0.39 ± 0.05e	0.137 ± 0.005d	38.02 ± 0.75f	13.42 ± 0.48a	129.01 ± 9.62a

Data are reported as mean ± standard deviation

Table 2.

Total phenolic (TPC) and tocopherol contents of raw and roasted hazelnuts samples. Different letters in the same column showed significant differences ($p<0.05$).

Samples	Roasting conditions	TPC (mg/100g d.w.)	Tocopherols (mg/100g oil)		
			α-tocopherol	*β-tocopherol*	*γ-tocopherol*
Raw		1245.27±25.19d	73.90±0.16a	2.01±0.38a	5.24±0.54a
LT 1	130°/40min	2017.27±119.40c	80.67±6.27a	1.53±0.31a	3.85±0.45a
LT 2	130°/50min	2218.25±116.17c	78.94±4.38a	1.65±0.18a	4.47±0.23a
LT 3	130°/60min	2031.49±207.91c	75.83±2.58a	1.70±0.10a	4.22±0.17a
HT 4	160°/20min	2998.84±38.65b	77.30±7.42a	1.48±0.20a	3.74±0.71a
HT 5	160°/25min	3429.52±106.80a	75.55±5.32a	1.63±0.37a	3.57±0.57a
HT 6	160°/30min	2927.81±199.16b	74.05±2.37a	1.55±0.20a	4.73±1.74a

Data are reported as mean (n=3) ± standard deviation.

Table 3. Tentative volatile compounds obtained by SPME-GC-MS of raw and differently roasted hazelnuts samples.

Compounds	R	LT1	LT2	LT3	HT4	HT5	HT6
Ketones							
acetone	129.8±13.9	209.9±82.3	202.1±28.3	208.6±21.3	215.9±38.3	169.7±36.6	231.3±51.0
2-butanone	n.d.	7.8±2.2	23.2±11.0	18.4±2.8	23.7±2.5	29.9±0.9	26.2±5.9
2-pentanone	2.6±0.4	4.7±1.8	5.7±0.7	5.6±0.7	6.5±0.1	n.d.	4.7±0.6
2,3-butanedione	n.d.	n.d.	n.d.	n.d.	21.4±3.8	36.6±7.7	26.1±2.5
3-methyl-2-pentanone	n.d.	48.8±5.6	74.3±20.0	48.6±1.1	62.0±3.7	75.3±9.0	48.2±7.6
1-(2-furanyl)-ethanone	n.d.	n.d.	n.d.	n.d.	3.9±0.5	6.3±0.3	3.3±0.1
3-hexanone	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.0±0.1
2,3-pentanedione	n.d.	17.2±3.8	22.1±6.2	17.2±2.2	35.9±6.7	50.3±6.4	47.7±5.6
4-ethyl-3-hexanone	n.d.	3.9±1.3	6.5±1.9	6.3±0.7	n.d.	n.d.	n.d.
3-penten-2-one	n.d.	54.8±5.6	77.7±15.2	69.4±0.8	68.8±4.6	95.5±5.2	76.3±2.1
cis-3,5-dimethyl-cyclohexanone	n.d.	n.d.	n.d.	n.d.	n.d.	12.0±2.7	9.8±3.0
2-heptanone	n.d.	n.d.	n.d.	3.1±0.9	n.d.	n.d.	5.7±0.0
(2E)-5-methyl-2-hepten-4-one	n.d.	49.4±4.1	44.0±5.8	63.6±2.7	67.9±6.0	93.3±2.5	49.8±1.2
3,5-dimethyl-4-heptanone	n.d.	14.2±0.7	23.0±3.1	28.7±3.7	29.6±4.4	40.9±1.2	23.5±2.1
3-methyl- 4-heptanone	n.d.	2.7±0.6	5.7±2.4	5.2±0.6	3.7±0.7	5.7±1.0	3.1±0.3
5-hydroxy-2-pentanone	n.d.	n.d.	n.d.	n.d.	n.d.	5.2±2.4	6.8±0.9
5-methyl-5-hexen-2-one	n.d.	1.9±1.3	2.9±0.6	2.6±0.2	1.3±0.9	4.3±1.4	n.d.
5-methyl-3-hexen-2-one	n.d.	n.d.	1.6±0.4	n.d.	n.d.	n.d.	n.d.
3-methyl-4-hexen-2-one	n.d.	n.d.	n.d.	n.d.	3.4±0.1	n.d.	n.d.

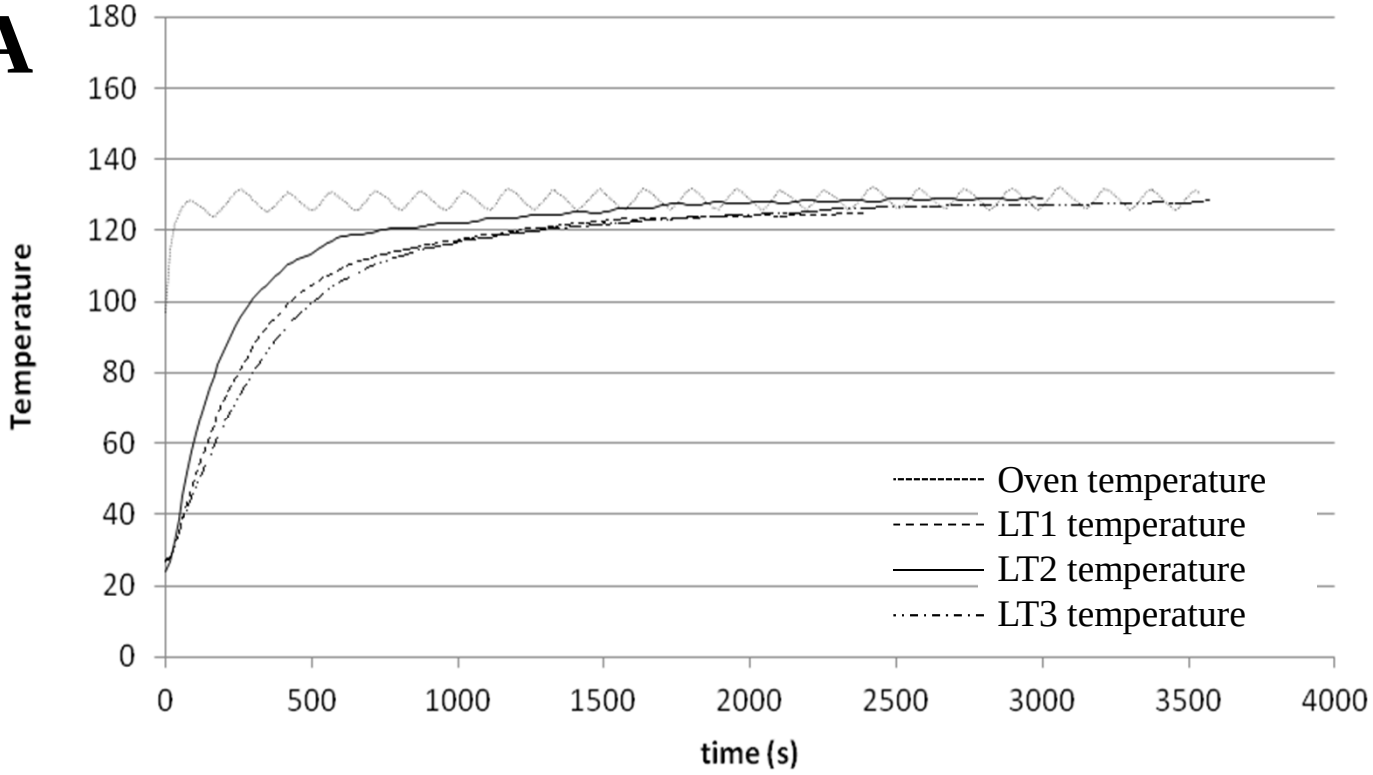
3-hydroxy-2-butanone	n.d.	11.7±0.3	16.7±3.4	14.5±0.9	37.3±6.2	49.3±4.4	35.9±3.6
4-hexen-3-one	n.d.	101.6±2.0	93.3±10.8	105.5±6.7	151.5±6.2	187.7±6.4	124.5±5.8
3-hepten-2-one	n.d.	3.8±0.4	4.6±1.5	3.7±0.3	2.3±0.5	3.3±0.2	1.7±0.4
1-hydroxy- 2-propanone	n.d.	13.4±1.0	15.8±1.7	15.2±0.2	43.6±1.5	132.7±7.9	185.2±5.6
1-hydroxy-2-butanone	n.d.	n.d.	1.5±0.1	0.9±0.1	2.8±0.2	4.5±0.9	5.4±0.0
1-(acetyloxy)-2- propanone	n.d.	2.0±0.2	4.4±0.4	4.4±0.3	8.5±0.3	12.9±0.8	18.4±3.3
trans,trans-3,5-heptadien-2-one	n.d.	11.0±0.4	15.0±1.7	12.4±1.2	18.5±0.7	26.4±0.3	12.6±2.7
2-hydroxy-2,4-dimethyl-3-pentanone	n.d.	3.5±0.1	4.0±0.5	4.0±0.7	n.d.	n.d.	n.d.
1-(2-furanyl)-ethanone	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8.4±0.8
butyrolactone	n.d.	1.2±0.0	2.5±0.4	3.7±0.0	9.6±0.8	17.6±2.2	24.2±0.8
1-(1H-pyrrol-2-yl)-ethanone	n.d.	n.d.	n.d.	n.d.	3.0±0.2	4.4±0.6	8.3±0.2
2,5-dimethyl-4-hydroxy-3(2H)-furanone	n.d.	n.d.	n.d.	n.d.	5.9±0.5	12.5±1.9	25.4±1.3
dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone	n.d.	n.d.	n.d.	n.d.	2.7±0.1	4.4±0.2	7.5±0.5
Aldehydes							
2-methylpropanal	n.d.	78.7±18.3	122.3±17.7	195.2±3.8	367.7±72.4	569.1±80.7	391.4±68.3
2-methylbutanal	1.7±0.0	263.6±56.9	323.1±13.2	523.3±16.0	834.0±43.1	1313.6±45.1	817.9±34.3
3-methylbutanal	1.6±0.0	356.4±90.1	431.0±55.5	556.9±35.7	747.8±50.8	994.2±5.8	466.6±7.0
pentanal	n.d.	n.d.	1.3±1.2	n.d.	n.d.	n.d.	5.8±0.2
2-butenal	n.d.	3.0±0.0	5.2±0.1	4.3±0.1	9.4±1.6	17.8±0.7	20.5±0.5
hexanal	11.1±0.3	16.7±3.7	27.2±4.5	19.3±0.5	26.3±2.8	12.8±1.0	15.5±2.1
(E)-2-hexenal	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4.4±0.3
2-methyl-(E)-2-butenal	n.d.	n.d.	1.6±0.4	2.3±0.2	n.d.	n.d.	n.d.

nonanal	7.4±0.6	n.d.	2.1±0.0	3.4±0.4	n.d.	n.d.	n.d.
furfural	n.d.	2.8±0.3	n.d.	n.d.	n.d.	208.7±16.1	401.0±21.7
benzaldehyde	n.d.	5.6±1.1	3.5±0.4	3.1±0.3	5.2±0.8	4.2±0.2	4.8±0.2
5-methyl-2-furancarboxaldehyde	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	11.9±0.6
benzeneacetaldehyde	n.d.	n.d.	3.6±0.9	1.8±0.3	7.0±1.6	7.0±2.0	4.3±0.1
Pyrazines							
pyridine	n.d.	n.d.	n.d.	4.7±1.0	10.8±0.9	15.8±0.6	22.9±2.7
methylpyrazine	n.d.	11.7±1.0	32.7±3.6	43.1±2.6	119.7±2.1	196.5±9.8	288.0±20.0
2,5-dimethylpyrazine	n.d.	19.2±3.1	34.8±1.2	45.2±0.6	145.7±3.7	170.2±0.8	220.0±6.8
2,6-dimethylpyrazine	n.d.	3.2±1.2	4.9±0.3	8.3±0.7	38.7±1.9	64.1±4.9	101.6±5.8
ethylpyrazine	n.d.	2.3±0.1	6.5±1.2	7.1±0.7	20.9±2.3	29.3±1.3	42.8±1.4
2-ethyl-6-methylpyrazine	n.d.	n.d.	1.7±0.2	2.8±0.2	9.9±0.9	14.9±1.4	27.2±3.2
2-ethyl-5-methylpyrazine	n.d.	7.2±1.7	9.7±0.3	10.3±2.1	30.9±2.5	32.7±2.7	38.0±5.7
trimethylpyrazine	n.d.	3.5±2.4	4.6±0.2	5.3±1.2	22.4±1.4	26.7±1.6	32.6±4.6
2-ethyl-3-methylpyrazine	n.d.	n.d.	n.d.	1.1±0.2	4.4±0.7	5.8±1.0	14.1±3.5
3-ethyl-2,5-dimethylpyrazine	n.d.	3.4±0.5	5.3±0.5	5.7±0.0	16.7±2.5	21.8±0.5	31.0±5.8
2,5-diethylpyrazine	n.d.	n.d.	0.9±0.0	1.5±0.4	2.4±0.3	n.d.	n.d.
2,3-dimethyl-5-ethylpyrazine	n.d.	n.d.	n.d.	n.d.	n.d.	2.0±0.3	5.5±1.0
2-ethenylpyridine	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.2±0.4
N-acetyl-4(H)-pyridine	n.d.	n.d.	n.d.	n.d.	3.2±0.1	5.4±0.3	8.2±0.4
Furans							
2-methylfuran	n.d.	n.d.	n.d.	n.d.	n.d.	4.6±0.3	4.7±0.8

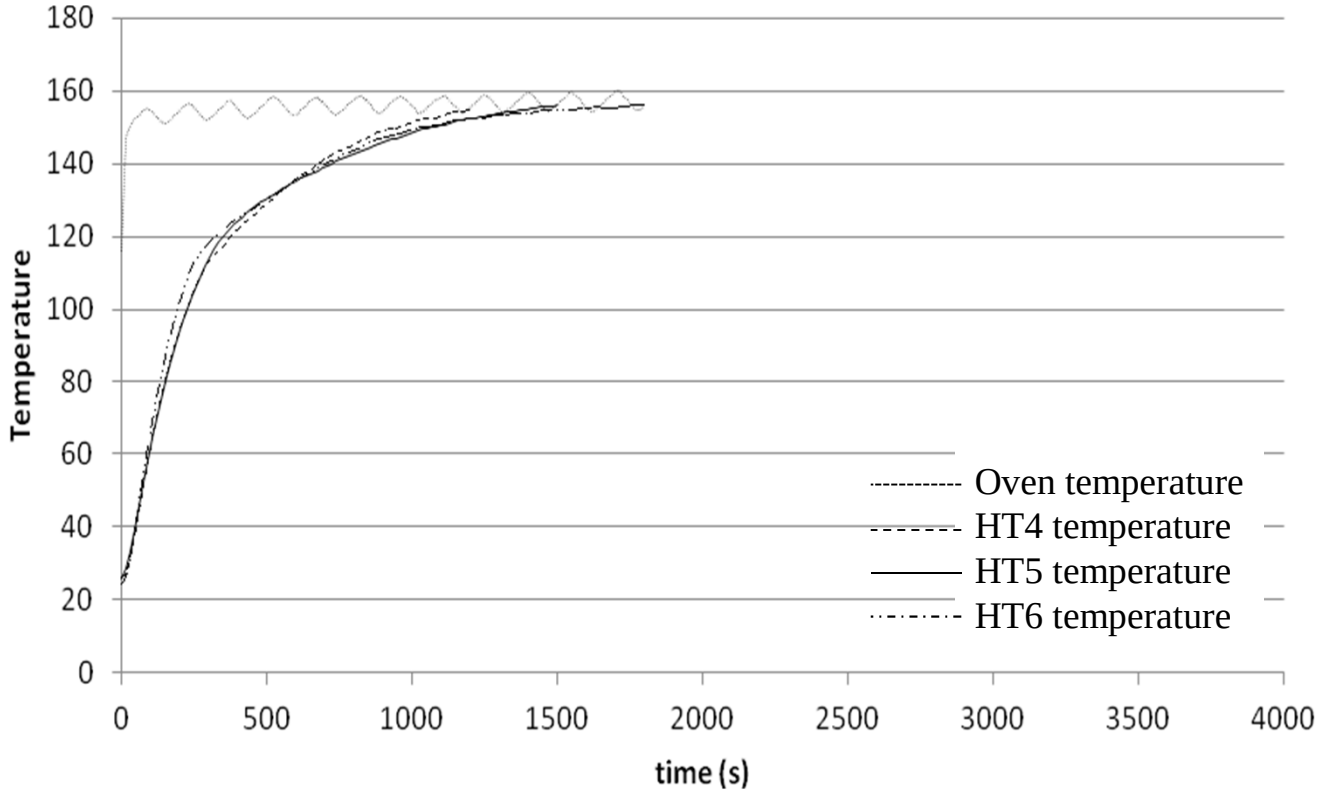
2,5-dimethylfuran	n.d.	5.3±0.1	8.3±0.4	9.0±0.1	8.8±0.6	16.4±2.8	22.0±0.2
2-ethyl-5-methylfuran	n.d.	3.5±0.3	3.4±0.3	5.0±0.9	n.d.	n.d.	n.d.
2,3,5-trimethylfuran	n.d.	7.6±0.5	6.7±0.1	6.2±0.3	7.1±0.4	7.6±0.4	6.9±0.6
2,3-dihydro-4-methylfuran	n.d.	n.d.	n.d.	n.d.	3.5±0.7	5.6±0.2	8.3±1.0
2-pentylfuran	n.d.	n.d.	1.1±0.3	0.9±0.1	4.0±0.3	2.4±0.1	4.7±0.0

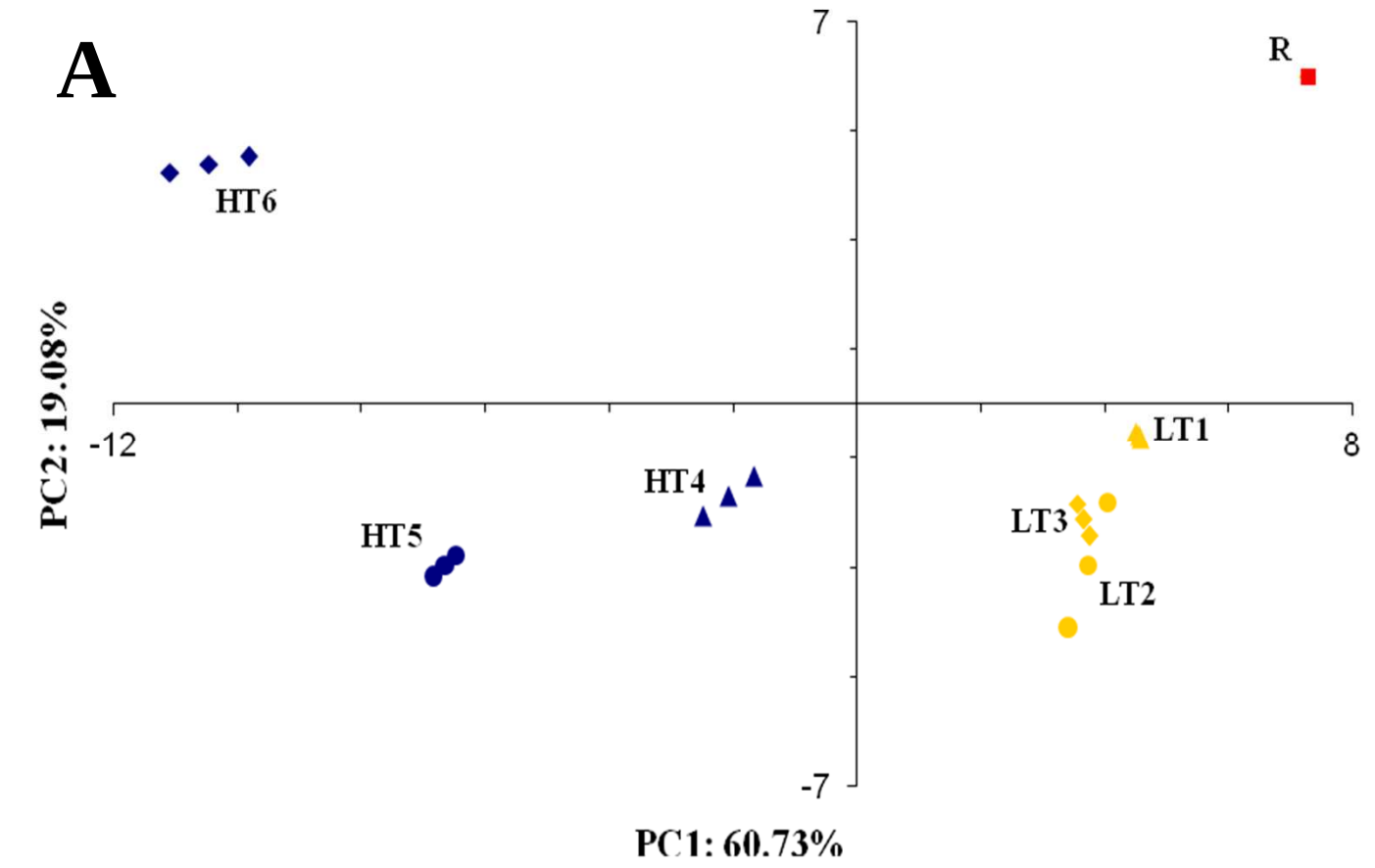
Areas (area x 10^5) are expressed as mean ± SD (*n*=3).

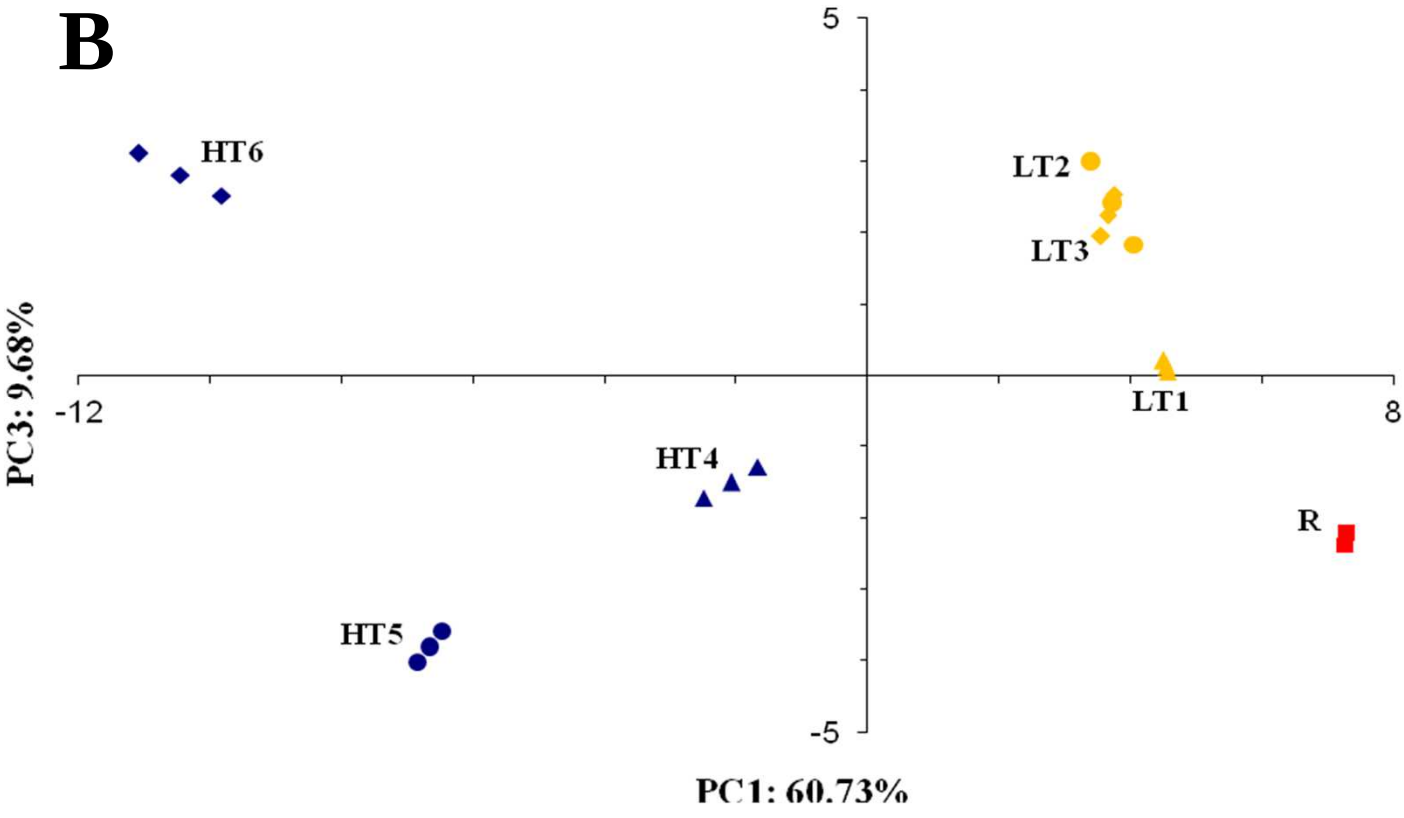
A


Temperature
180
160
140
120
100
80
60
40
20
0
0
500
1000
1500
2000
2500
3000
3500
4000
time (s)
Oven temperature
LT1 temperature
LT2 temperature
LT3 temperature

B


Temperature
180
160
140
120
100
80
60
40
20
0
0
500
1000
1500
2000
2500
3000
3500
4000
time (s)
Oven temperature
HT4 temperature
HT5 temperature
HT6 temperature


A
7
R
HT6
PC2: 19.08%
-12
8
LT1
HT4
LT3
HT5
LT2
-7
PC1: 60.73%


B
5
HT6
LT2
LT3
PC3: 9.68%
-12
LT1
8
HT4
R
HT5
-5
PC1: 60.73%

Highlights:

- Different temperature/time roasting conditions were applied to hazelnuts
- The phenolic content increased in all roasted samples
- The hazelnuts roasted at 160 °C demonstrated a phenolic content 49.3% higher than ones roasted at 130 °C
- In all samples α-tocopherol was the predominant tocol
- In samples roasted at 130 °C and 160 °C were found 79 and 102 volatile compounds