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Effects of different roasting conditions on physical-chemical properties of Polish hazelnuts (*Corylus avellana* L. var. Katalonski)

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

3

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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

53

54 **1. Introduction**

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

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

72 Roasting process is carried out to remove the pellicles of kernels, inactivate enzymes, destroy
73 microorganisms and reduce water activity (Özdemir, Seyan, Bakan, İter, Özey & Devres,
74 2001); moreover, roasting is used to improve the colour, the crispy texture and the flavour of
75 the product (Burdack-Freitag & Schieberle, 2010). The thermal treatment applied during
76 roasting processes leads to physical changes such as dehydration (Amaral, Casal, Seabra &
77 Oliveira, 2006), colour modifications (Alamprese, Ratti & Rossi, 2009), biochemical changes
78 including lipid structure modification (Amaral et al., 2006) and Maillard reactions that give

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

81 The roasting conditions generally used for hazelnuts are in a range from 100 to 160 °C for 10
82 to 60 minutes (Donno et al., 2013). Ciemniowska-Żytkiewicz, Bryś, Bryś, Sujka & Koczoń
83 (2014) roasted hazelnut *Kataloński* variety at three temperatures (100, 130, 160°C), of which
84 130 and 160 °C were reported as the most suitable for hazelnut sample final characteristics.
85 Roasted hazelnuts are used in food production such as chocolate spreads, ice creams, cereal
86 bars, cookies, etc. (Cucu, Platteau, Taverniers, Devreese, de Loose & de Meulenaer, 2011).

87 Different authors studied the influence of roasting conditions on physical-chemical properties
88 of hazelnuts. Ciemniowska-Żytkiewicz' et al. (2014) showed a decrease of moisture content
89 according to the temperature/time conditions and a change of hazelnuts' colour with a
90 decrease of L^* and a^* values compared to raw samples. Schmitzer, Slatnar, Veberic, Stampar
91 & Solar (2011) and Pelvan et al. (2012) have observed a loss in phenol content of about
92 66.3% in roasted hazelnuts in respect to raw ones, due to the removal of the skin which
93 contains the majority of phenols. Some authors investigated also the trend of tocopherols
94 during roasting: Schlörmann et al. (2015) showed a decrease of α and β -tocopherols after
95 roasting treatment of about 34% and 40%, respectively, whereas Amaral et al. (2006) found
96 only a reduction of 9% of α -tocopherol content at roasting conditions of 185 °C/15 min, as
97 compared to raw hazelnuts. Finally, Alasalvar, Shahidi & Cadwallader (2003a) have
98 compared the volatile compositions of raw and roasted hazelnuts (165 °C/25 min). After
99 roasting, hazelnut volatile profile was more concentrated and rich in new other compounds,
100 not present in the raw samples.

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

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

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

112

113 **2. Materials and Methods**

114

115 *2.1. Chemicals*

116 All the solvents and reagents for phenolic compounds and lipid extraction were from Sigma
117 Aldrich (Saint Louis, MO, USA). Folin Ciocalteu's reagent was purchased from Merck
118 (Darmstadt, Germany) and Na₂CO₃ for the determination of total phenolic content was from
119 BDH AnalaR® (Poole, England). All the solvents for the determination of tocopherols were
120 supplied by VWR Prolabo Chemicals (Dublin, Ireland).

121

122 *2.2. Samples*

123 *Kataloński* variety hazelnuts (*Corylus avellana* L.) were obtained from an orchard located in
124 the south of Poland (Jankowice, Pszczyna 50°0' 5" N 18°59' 18" E) in 2013. Hazelnuts
125 were collected at complete maturity, sun-dried for 3 days at 20-25 °C and stored with shell at
126 4 °C until the analysis.

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

130

131 *2.3. Roasting of hazelnuts*

132 Approximately 50 g of shelled hazelnuts were roasted in a lab-scale ventilated oven (Vismara,
133 Italy) at different time and temperature conditions: 130 °C (Low Temperature, LT) for 40 (1),
134 50 (2) and 60 (3) minutes, and 160 °C (High Temperature, HT) for 20 (4), 25 (5) and 30 (6)
135 minutes. Each roasting protocol was carried out three times.

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

143

144 *2.4. Moisture and water activity determination*

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

148 Water content (%) was evaluated on ground hazelnut samples in an oven at 105 °C until
149 constant weight was reached. For each sample, 3 replicates of 3 g weighted were dried
150 (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):

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

163 where,

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

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

173 methanol-water (1/1 v/v). Each extraction was carried out two times for each set of roasting (n
174 = 6 for roasting condition) and the extracts were stored at -18 °C until use.

175

176 *2.7. Determination of total phenolic content*

177 The total phenolic content (TPC) of the extracts was assessed by means of the Folin-Ciocalteu
178 method (Singleton & Rossi, 1965). Briefly, 100 μ L of each extract was shaken with 500 μ L
179 Folin–Ciocalteu reagent and 6 mL of distilled water. Two millilitres of 15% Na₂CO₃ was
180 added and the mixture was shaken once again for 30 seconds. Finally, the solution was
181 brought up to 10 mL by adding distilled water. After 2 h, the absorbance at 750 nm was
182 measured using glass cuvettes. The phenolic content was calculated on the basis of the gallic
183 acid calibration curve (from 25 to 1000 μ g/mL). Absorptions were measured in 2 replicates
184 for each extract ($n = 12$ for roasting condition) and the results were expressed as mg/100g of
185 hazelnuts d.w.

186

187 *2.8. Extraction of lipid fraction*

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

193

194 *2.9. Tocopherols analysis*

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

203

204

205 2.10. SPME-GC-MS analysis

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

210 About 3 g of ground hazelnut were weighed into a 10 mL amber vial, crimped with
211 aluminium caps equipped with a septum. The samples were equilibration at 40 °C for 30
212 minutes; a 2 cm x 0.11 µm (i.d.), 50/30 µm divinylbenzene/carboxen/polydimethylsiloxane
213 (DVB/Carboxen/PDMS) SPME fiber (Supelco, Bellefonte, PA, USA) was then inserted
214 through the septum into the vial at 40 °C for another 10 minutes; vial penetration depth was
215 20 mm. Afterwards, the SPME fiber was desorbed at 240 °C for 7 minutes in the split mode.
216 An Rtx-Wax fused-silica capillary column (30 m x 0.25 mm i.d. x 1.0 µm f.t.) (Phenomenex,
217 Torrance, CA, USA) was used for the chromatographic separation. The oven was
218 programmed from 40 °C (kept for 10 minutes) to 200 °C at 3 °C/min and maintain it for 3
219 minutes, then increased from 200 °C to 240 °C at 10 °C/min and kept at the final temperature

220 for 5 minutes. The injector, transfer line and the ion source temperatures were set at 240 °C,
221 240 °C and 200 °C respectively. Helium was used as the carrier gas at an inlet pressure,
222 constant flow rate of 1.5 mL/min; the split ratio was 1:10.

223 The filament emission current was 70 eV. A mass range from m/z 30 to 250 was scanned from
224 3.5 to 70 minutes. The acquisition was carried out in Total Ion Current (TIC) mode, using the
225 GCMS solution software, version 2.50 SU1 (Shimadzu, Tokyo, Japan). Each extraction was
226 carried out two times for each set of roasting ($n = 6$ for roasting condition).

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

230

231 *2.11. Statistical Analysis*

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

236

237

238 **3. Results and discussion**

239 The thermal profiles of oven and hazelnuts samples obtained during roasting processing at
240 130 °C (LT) and 160 °C (HT) for different times are respectively reported in Figures 1A and
241 1B. As show in Figure 1A the temperature inside roasted hazelnuts reached a plateau around
242 130 °C between 25 to 30 minutes of treatment, whereas roasted hazelnuts at 160 °C showed
243 an increase of temperature inside the product more abruptly, reaching 130-135 °C during the
244 first 8-10 minutes (Figure 1B).

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

248

249 *3.1. Moisture, water activity and colour*

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

255 The moisture of raw kernels was 4.86% on average; this result agreed with those reported in
256 literature (Amaral et al. 2006; Saklar et al., 2001) for different hazelnut varieties. As expected,
257 moisture content decreased significantly in the samples proportionally to the intensity of
258 applied process in terms of temperature/time conditions. The sample roasted for 30 minutes at
259 160 °C underwent the highest water loss.

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

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

270 colour attribute is analogous to the colour observation made by the operator (Moss & Otten
271 1989; Ozdemir & Devres 2000). In this work browning index (BI) was also calculated, in
272 order to more precisely evaluate the overall colour changes in hazelnut samples during
273 roasting.

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

281

282

283 *3.2. Total phenolic content (TPC) and tocopherols composition*

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

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

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

300 Results obtained within this study were in disagreement with those reported by Pelvan et al.
301 (2012) and Schmitzer et al. (2011) where authors obtained a lower TPC value in hazelnuts
302 after roasting treatment; indeed Schmitzer and co-workers observed that the skin removal and
303 applied roasting conditions (15 min at 140° C) affected the total phenolic content negatively.
304 Several authors (Shahidi et al., 2007; Alasalvar, Karamać, Kosińska, Rybarczyk, Shahidi &
305 Amarowicz, 2009b; Locatelli, Travaglia, Coisson, Martelli, Stévigny & Arlorio, 2010)
306 affirmed that the skin has a key role in the determination of total phenolic content containing
307 the majority of them. The presence of skin in a part of *Kataloński* variety hazelnuts roasted in
308 these trials could be contributed to significantly affecting the obtained TPC levels.

309
310 The individual tocopherols identified are shown in Table 2; the results were in agreement with
311 literature results (Ciemniewska-Żytkiewicz' et al., 2015b, Alasalvar et al., 2003b; Amaral et
312 al., 2006); in all samples α -tocopherol was the predominant compound followed by γ -
313 tocopherol and β -tocopherol.

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

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

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

325

326 3.3. Volatile compounds

327 SPME-GC-MS analysis identified 22 compounds in raw hazelnuts, 79 in hazelnuts roasted at
328 130°C and 102 in those roasted at 160°C. Compounds identified were ketones, aldehydes,
329 pyrazines, furans, aromatic hydrocarbons, alcohols, terpenes and acids. In particular,
330 pyrazines, terpenes, pyrroles, furans and acids were identified only in roasted hazelnuts. As
331 reported by Alasalvar et al. (2003a) the compounds more responsible of roasted hazelnuts
332 aroma are ketones, aldehydes, pyrazines and furans.

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

336

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

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

344 Rubiolo, Sgorbini & Liberto, 2013) to be the compound that contributes to the typical nutty-
345 roasty and hazelnut-like aroma of this nut. In samples roasted at both tested conditions, its
346 concentration had no linear trend; in fact decreased from LT1 to LT2 samples and from HT5
347 to HT6 samples, moreover increased from LT2 to LT3 samples and HT4 and HT5. The
348 sample that had the highest concentration of filbertone was HT5. In different studies
349 (Burdack-Freitag & Schieberle, 2012; Kiefl & Schieberle, 2013), only the concentration of
350 filbertone increased after roasting. These differences show that the concentration of this
351 compound can depend not only on conditions of roasting process but also on variety and the
352 fibrous skin presence. Moreover, other ketones play an important role in hazelnut aroma, like
353 3-penten-2-one that is responsible for fruity odour (Langourieux, Perren & Escher, 2000) and
354 2,3-pentanedione, a sugar degradation product responsible for sweet odour (Ho & Carlin,
355 1989). Concentration of both compounds increased in HT samples, in particular HT5 had the
356 highest contents of them, whereby fruity and sweet odours increased after roasting process in
357 160 °C significantly, which is expected by consumers.

358
359 *Aldehydes.* A total of 11 aldehydes were found in both LT and HT samples (Table 3).
360 According to Alasalvar et al. (2003a) the predominant aldehydes were 2-methylpropanal, 2-
361 methylbutanal and 3-methylbutanal. According to Burdack-Freitag & Schieberle (2012), the
362 concentration of these compounds, in particular of 3-methylbutanal, increased after roasting
363 causing simultaneously the increase of fruity, malty and chocolate-like odors (Alasalvar et al.,
364 2003; Burdack-Freitag & Schieberle, 2010). As reported for ketones, the HT5 sample was the
365 richest in these three aldehydes among all samples tested. Other aldehydes, 2-methyl-(E)-2-
366 butenal and nonanal were identified only in LT samples, however the (E)-2-hexenal and 5-
367 methyl-2-furancarboxaldehyde were present only in HT samples.

368

369 *Pyrazines*. A total of 11 pyrazines were detected in LT samples, whereas 14 compounds in
370 HT samples, in particular, 2,3-dimethyl-5-ethylpyrazine, 2-ethenylpyridine and N-acetyl-
371 4(H)-pyridine were present only in HT samples. These compounds, originated by Maillard
372 reaction from free amino acids and monosaccharides, contribute to nutty and roasty aroma
373 (Kiefl, Pollner & Schieberle, 2013a). According to Alasalvar et al. (2003a) the most abundant
374 compounds were 2,5-dimethylpyrazine and methylpyrazine; moreover, herein, 2,6-
375 dimethylpyrazine was one of major compound present in roasted hazelnut as well. The
376 concentration of all pyrazines increased with increasing roasting time, as reported in Table 3.
377 The highest content of these three pyrazines was detected in HT6 followed by HT5 sample.
378 Kiefl et al. (2013a) in their study observed the same trend, where pyrazines were rapidly
379 formed during roasting and they were correlated with aroma of hazelnuts and, in general, of
380 processed food.

381
382 *Furans*. As reported for pyrazines, also furans are originated from Maillard reaction
383 (Alasalvar et al., 2003a). Şenyuva & Gökmen (2007) reported that the formation of furans in
384 hazelnuts during heat treatment increased at temperature exceeding 120 °C. In this study,
385 furans were absent in raw hazelnuts and a total of 4 and 5 furans were detected in LT and HT
386 samples, respectively. 2-ethyl-5-methylfuran was present only in LT samples, however, 2-
387 methylfuran and 2,3-dihydro-4-methylfuran were identified only in HT samples. 2,5-
388 dimethylfuran was the most abundant compound in LT3 and HT6, in agreement with results
389 obtained by Alasalvar et al. (2003a), followed by 2,3,5-trimethylfuran that was higher in HT5
390 samples.

391
392 Figure 2A shows a PCA plot obtained with the areas of single volatile compounds, with
393 60.73% of the variation accounted for PC1 and 19.08% accounted for PC2. The figure shows
394 a good separation between hazelnut samples, in particular raw hazelnuts (R) has been well

395 separated from LT and HT ones. Raw sample, that had positive scores according to both PC1
396 and PC2, was discriminated from the other samples for the nonanal. PC1 discriminated HT
397 samples, that exhibited negative scores and LT samples that had positive scores. 2-pentanone,
398 4-ethyl-3-hexanone, 3-hepten-2-one, 2-hydroxy-2,4-dimethyl-3-pentanone, hexenal, 2-
399 methyl-(E)-2-butenal, 2,5-diethyl-pyrazine, 2-ethyl-5-methyl-furan were responsible for PC1
400 discrimination of the samples. For PC2, only R and HT6 samples exhibited positive scores, all
401 the other samples had negative scores. Some compounds responsible for this discrimination
402 were 2,5-dimethyl-4-hydroxy-3(2H)-furanone, dihydro-3-hydroxy-4,4-dimethyl-2(3H)-
403 furanone, butyrolactone, 2-ethyl-6-methyl-pyrazine, N-acetyl-4(H)-pyridine, methyl-pyrazine,
404 ethyl-pyrazine, 3-ethyl-2-dymethyl-pyrazine and 2,3-dihydro-4-methyl-furan.

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

413

414 **4. Conclusions**

415 This study confirms that time and temperature used for hazelnuts roasting treatment can
416 deeply influence their final quality. Obtained results show that the low temperature and short
417 roasting time are not always the most appropriate way to obtain the best product, in terms of
418 total phenolic content, tocopherols and volatile profile. In fact the highest concentration of
419 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

428

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434

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436

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- 544

545

546 **Captions of figures**

547

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

551

552

553 **Figure 2.** PCA score plots (PC1 x PC2, figure 2A) (PC1 x PC2, figure 2B) of volatile
554 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) | | |
|---------|---------------------|--------------------|---------------------------------------|--------------------------------------|---------------------------------------|
| | | | <i>α-tocopherol</i> | <i>β-tocopherol</i> | <i>γ-tocopherol</i> |
| 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 |

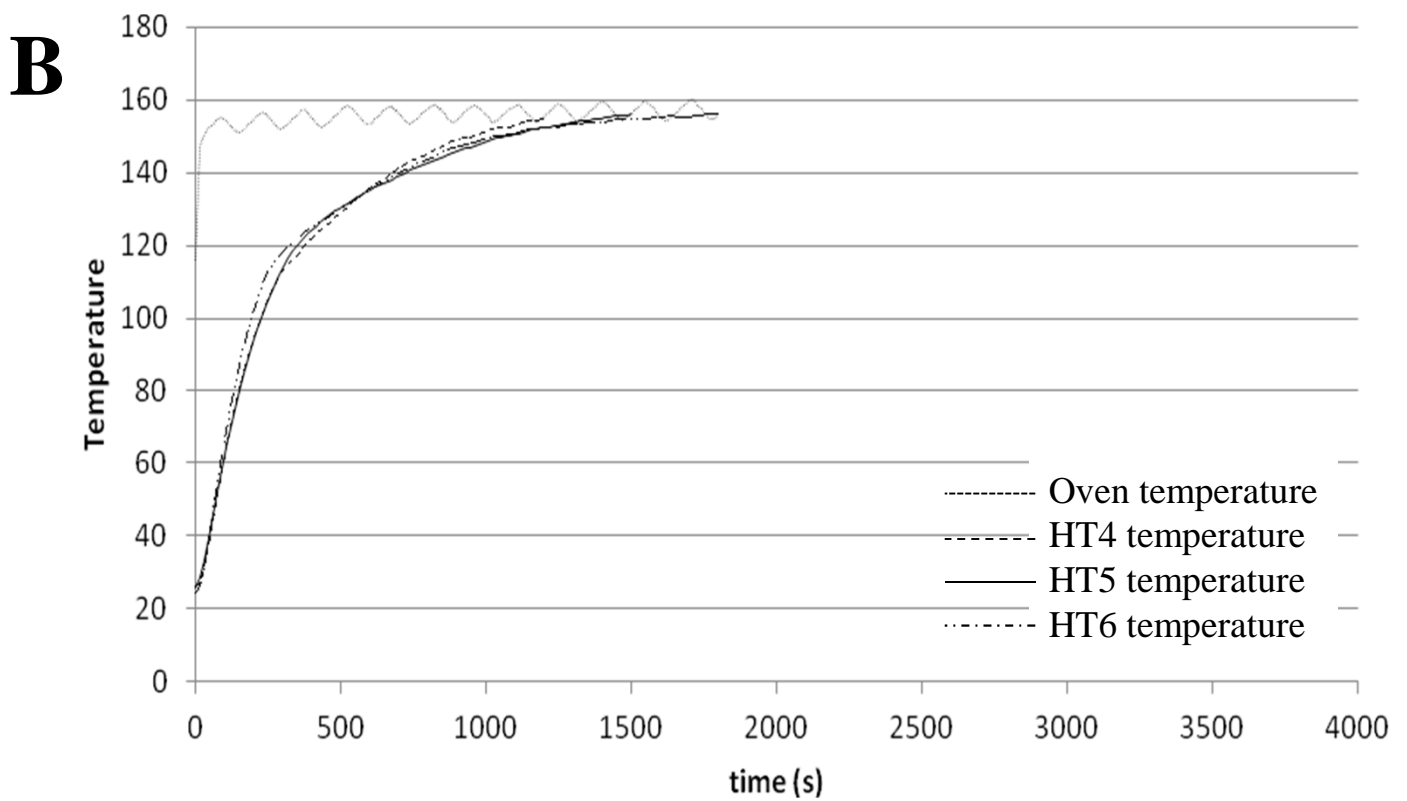
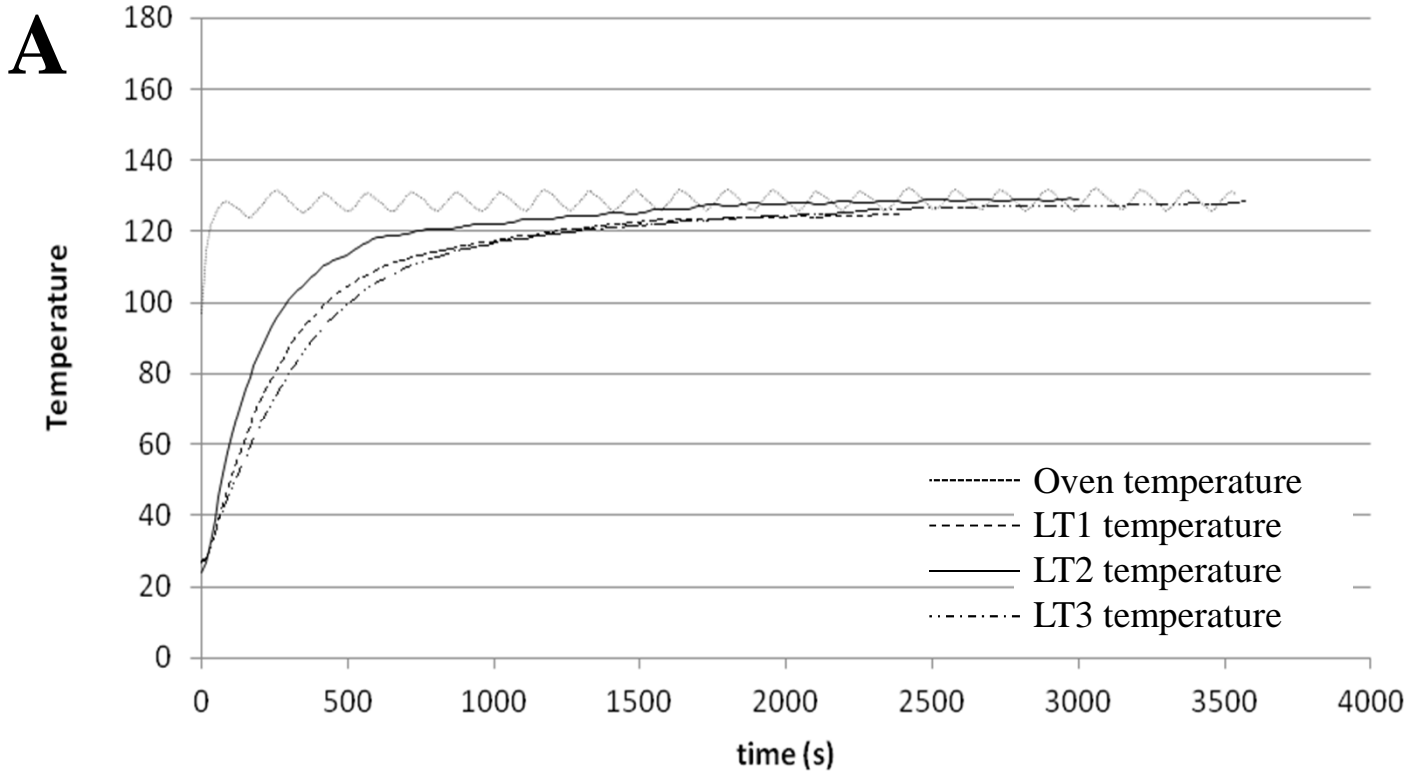
Aldehydes

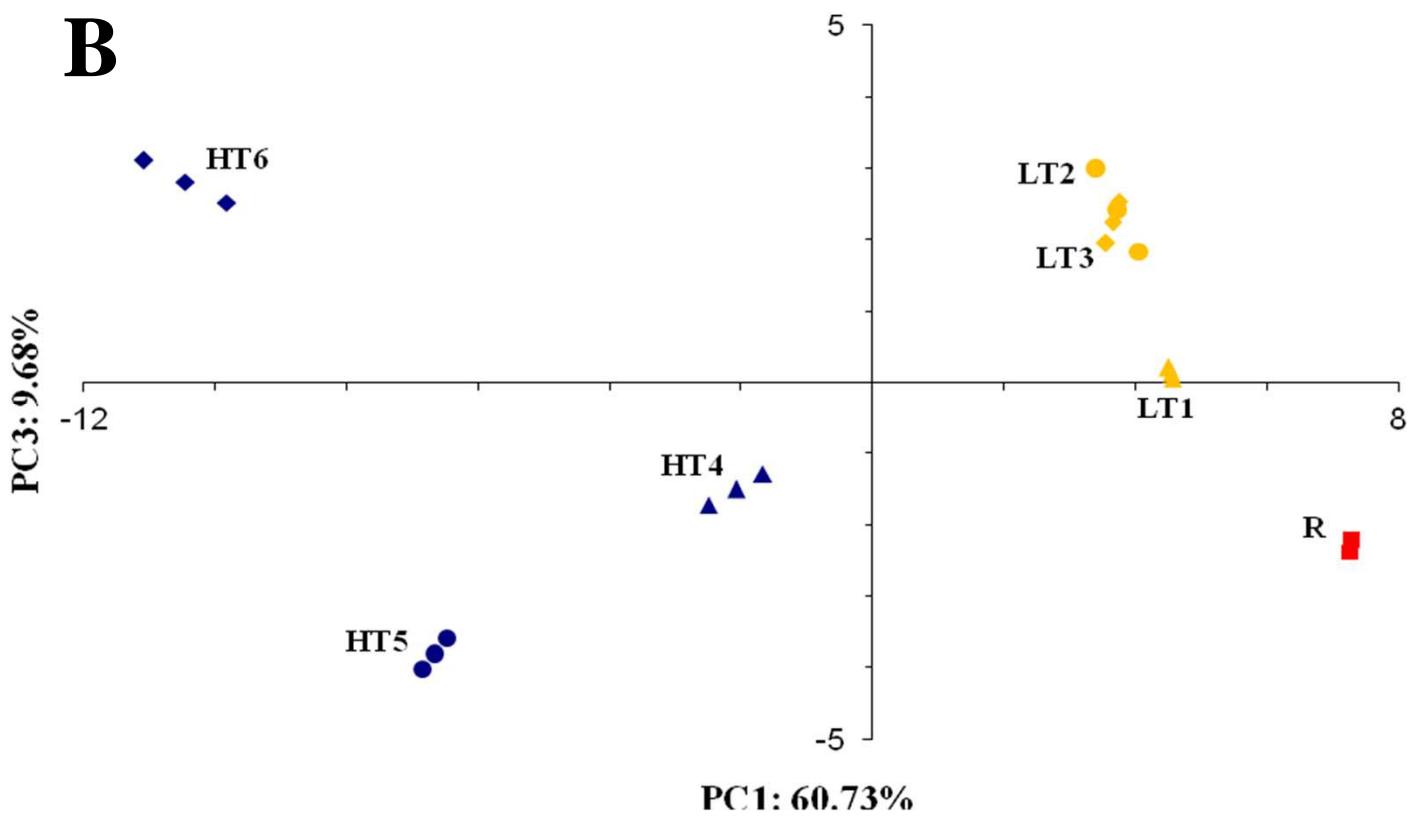
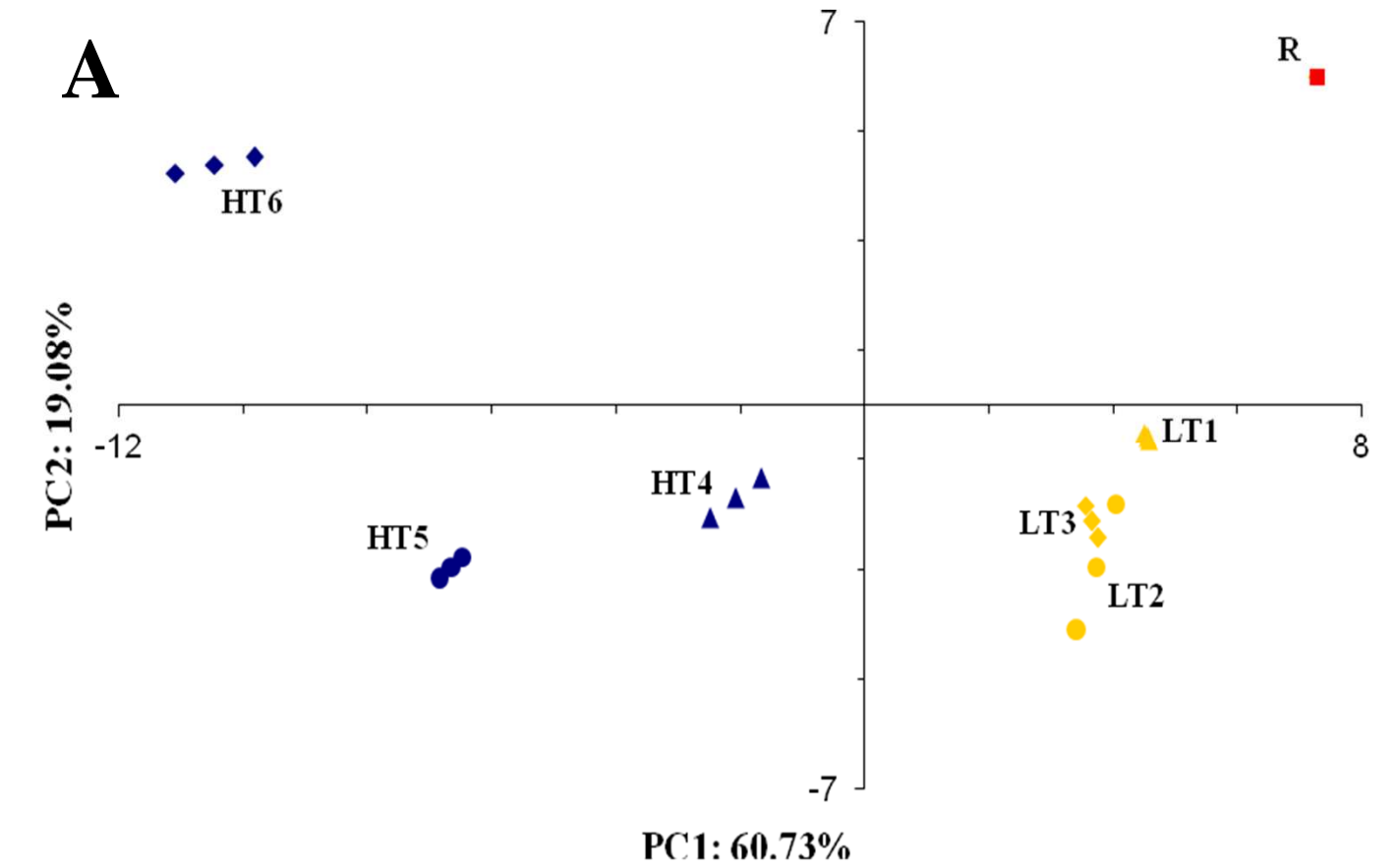
| | | | | | | | |
|------------------------|----------|------------|------------|------------|------------|-------------|------------|
| 2-methylpropanal | n.d. | 78.7±18.3 | 122.3±17.7 | 195.2±3.8 | 367.7±72.4 | 569.1±80.7 | 391.4±68.3 |
| 2-methylbutanal | 1.7±0.0 | 263.6±56.9 | 323.1±13.2 | 523.3±16.0 | 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 10⁵) are expressed as mean ± SD (n=3).





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