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Roasting of hazelnuts. Role of oil in colour development and hydroxymethylfurfural formation

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Abstract

The roles of oil, hexanal and sucrose were investigated in colour development and HMF formation during roasting of hazelnuts. The highest HMF levels and the deepest browning extension were in crushed hazelnuts and the lowest HMF and browning were in defatted or partially defatted samples. The addition of hexanal, at 175 °C, increased the HMF level. This was particularly evident in samples with sucrose. Moreover samples totally reintegrated in oil did not reach (after roasting) the same HMF level and colour as the corresponding crushed samples.

Keywords: Hazelnuts; Browning; HMF; Hexanal; Sucrose

1. Introduction

Hazelnuts, due to their organoleptic characteristics, constitute one of the most important raw materials for the pastry and chocolate industry. Only a small amount, (8–10%) of the annual hazelnut production is consumed as nuts. Quality parameters used by industries focus on the variety of the hazelnuts, defects of the kernels and the amount and characteristics of the hazelnut oil.

Lipid oxidation has a very strong effect on shelf life and sensory characteristics of hazelnuts. It depends on many factors such as the concentrations of unsaturated fatty acids enzymatic activity, mineral composition and the presence of antioxidants (Özdemir & Devres, 1999). The ratio unsaturated/saturated fatty acid has been proposed to predict hazelnut shelf life (Pershern, Breene, & Lulai 1995). Prolonged storage of hazelnuts induces the formation of volatile off-flavours, alkanals, 2-alkenals and alkanoic acids. Hexanal and octanal are the most important lipid oxidation products, reaching up to tenfold their starting concentrations (Kinderlerer & Johnson, 1992). Roasting is the heat treatment for inducing development of the typical colour, taste and flavour of hazelnuts; it also changes the chemical composition, modifying nutritional value and shelf-life (Özdemir & Devres, 1999; Özdemir, Ackurt, Yildiz, Biringen, Gurcan, & Loker, 2001). The roasting process is usually conducted empirically and based on experience. This might be ascribed to the very complex network of chemical reactions summarised under the names of Maillard reactions (MR). In order to achieve a systematic approach to roasting some recent studies have been carried out on the optimisation of roasting parameters and colour development (Özdemir & Devres, 1999, 2000; Perren & Escher, 1999).

Other studies carried out on crushed and crushed-defatted hazelnuts, respectively, have shown different extensions of the browning reaction and different antioxidant activities during roasting and/or subsequent storage (Severini, Romani, & Lerici, 1995).

The majority of studies on Maillard reactions are conducted in aqueous model systems containing two reactive components, comprising a sugar and an amino compound. Studies involving three-component Maillard systems, for example, sugar, amino acid and lipid, are less common and they have been focussed on flavour rather than colour generation (Farmer & Mottram, 1990, 1992; Farmer, Mottram, & Whitfield, 1989).

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Vasiliauskaite and Wedzicha (1997) investigated the role of formaldehyde, produced during the Strecker degradation of glycine, in a glycine/glucose system. Formaldehyde inhibited browning, and the melanoidins formed in its presence were different from those formed in absence of formaldehyde. Fallico and Ames (1999), studying an aqueous glucose/phenylalanine model system, heated in an autoclave at 140 °C, found that the addition of hexanal and/or Fe^{2+} to the system significantly inhibited colour development and led to an increase of HMF.

In the past decade, the recovery of hazelnut oil and alternative uses have been proposed (Casini & Tortorella, 1991; Contini, De Santis, Frangipane, & Anelli, 1994). Furthermore, defatted hazelnut flour has been proposed (after a partial oil reintegration) as a low fat raw material for the pastry industry (Contini et al., 1994).

As colour development is one of the most important parameters during roasting of hazelnuts, and HMF level is often used as index of browning extension, the aim of this paper is to investigate the development of these parameters in hazelnuts after roasting. Moreover, the roles of oil, a lipid oxidation product (hexanal) and the addition of sucrose were investigated.

2. Materials and methods

2.1. Samples

All the experiments were conducted on hazelnuts, variety *Mortarella*, from Novi Ligure (Alessandria, Italy). Two kilogrammes of hazelnuts were blanched for two minutes in boiling water and peeled, dried and crushed in a domestic electric mixer. In order to obtain a homogeneous sample, the crushed hazelnuts were passed through two sieves with 2000 μm pores (mesh/n10) and 1000 μm pores (mesh/n18), keeping the portion isolated between the two sieves. The crushed hazelnuts were divided into two parts; the first one was used as a reference sample and the second to extract the oil.

2.2. Oil extraction and defatted crushed hazelnuts

The oil extractions were carried out on 100 g portions of crushed hazelnuts using 500 ml of petroleum ether (40–60, Merck, Milan) under stirring for 1 h. The extraction was repeated three times, using 300 ml of petroleum ether each time. The ether extracts were combined and the solvent was distilled under vacuum at low temperature (<20 °C). Both oil and defatted crushed hazelnuts (DCH) were kept in desiccators under vacuum, up to 24 h to remove the last solvent traces. Moisture and oil content were determined on the reference sample according to AOAC (1990).

2.3. Roasting and colour determination

Table 1 lists codes and compositions of samples used for roasting. All percentages were calculated on DCH weight and samples were prepared in duplicate. Samples with hexanal were obtained by adding, to DCH, oil containing increasing concentration of hexanal (1.3–104 ppm).

Roasting experiments were carried out in a ventilated oven at 150 °C for 60 min, and at 175 °C for 30 and 60 min, respectively.

Colour parameters, L^*, a^*, b^*, C and h, were determined on roasted samples using a hand colorimeter

Table 1
Codes and compositions of samples

Sample	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W
Crushed hazelnuts (reference sample)	x									x													
Defatted crushed hazelnuts (DCH)		x	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x	x	x	x	x	x
10% water			x																				
10% hazelnut oil				x																			
20% hazelnut oil					x																		
30% hazelnut oil						x														x			
40% hazelnut oil							x																
60% hazelnut oil								x															
30% paraffin oil									x														
10% hazelnut oil (1.3 ppm hexanal)												x											
10% hazelnut oil (12.5 ppm hexanal)													x										
10% hazelnut oil (104.3 ppm hexanal)														x									
30% hazelnut oil (1.3 ppm hexanal)															x								
30% hazelnut oil (12.5 ppm hexanal)																x					x		
30% hazelnut oil (20.9 ppm hexanal)																	x					x	
30% hazelnut oil (33.4 ppm hexanal)																		x					x
30% hazelnut oil (104.3 ppm hexanal)																			x				
30% sucrose										x	x									x	x	x	x

(Nippon Denshoku, NR 3000), using D65/10 as illuminant, and a white tile as reference. Each value was the average of three readings.

2.4. HMF extraction and HPLC determination

HMF was extracted from roasted samples with 50 ml of ethyl acetate (Merck, Milan) keeping the sample under stirring for 10 min. The extraction was repeated three times. The ethyl acetate extracts were combined, filtered and then distilled under vacuum at low temperature (<20 °C). The concentrated extracts were transferred to a volumetric flask, diluting up to 3 or 10 ml with ethyl acetate, for samples roasted at 150 or 175 °C, respectively, and injected into the HPLC.

The HPLC apparatus was a Varian 9012Q equipped with a diode array detector (Varian, Star 330). The HPLC column was a Merck Lichrospher, RP 18, 5 μm, 250×4 mm, fitted with a guard cartridge packed with the same stationary phase (Merck, Milan) and thermostatted at 30 °C. The mobile phase was isocratic up to 10 min: 95% water at 2% of acetic acid (A) and 5% methanol (B), then the column was washed with methanol (15 min). At 30 min, the apparatus was returned to its starting conditions (95% A, 5% B). The flow rate was 0.7 ml/min. All the solvents were HPLC grade (Merck, Milan). The injection volume was 20 μl and the chromatograms were monitorated at 285 nm. Spectra of peak were collected from 220 to 450 nm.

HMF was identified by spiking the peak of HMF extracted from roasted samples with the HMF standard (Sigma-Aldrich, Milan), and by comparison of the UV spectra of the HMF standard with that extracted from roasted samples. The amount of HMF was determined using an external calibration curve, measuring the signal at $\lambda = 285$ nm. The HMF concentration in all samples, including sample A (60% of oil), was expressed as mg/kg of DCH.

3. Results and discussion

Table 2 lists HMF levels and colour parameters of roasted samples at 150 °C for 60 min. Roasting of sample no. 1 led to the production of 8 mg/kg of HMF while, in the corresponding defatted samples (no. 2), only 2.2 mg/kg of HMF was found. As the reference sample (no. 1) had about 10% of moisture, in order to investigate the effect of water on HMF formation, the same amount of water was added to DCH (sample no. 3). The HMF concentration was 2.9 mg/kg. The addition of oil to the DCH induced an increase of HMF (samples 4–7) with respect to the defatted sample.

Significant differences, also, in colour parameters between the reference sample and partially or totally defatted ones were found (Table 2). The reference sample (no. 1) was the darkest as shown by the lowest L^* values (61.93) and the highest $a*$ value (9.2) according to literature data (Mastrocola & Munari, 2000). In the other samples, L^* ranged between 76.3 and 84.4, while a^* ranged between 3.8 and 6.3. Sample no. 1 also had the highest b^* and C values (36.8 and 38.0, respectively), while it had the lowest h value (76.0), confirming the deepest colouration of this sample. Sample no. 3 showed a different colour from both sample 2 and samples 4–7, indicating that the added water induced a little increase in HMF formation, but a much more influence on colour parameters.

Table 2
HMF concentration and colour parameters in roasted samples at 150 °C for 60 min

No.	Samples[a]	mg HMF/kg DCH[b]	L^*	a^*	b^*	C	h
1	A	8.0 (0.01)	61.9	9.2	36.8	38.0	76.0
2	B	2.2 (0.38)	82.3	3.8	29.0	29.3	82.6
3	C	2.9 (0.01)	76.3	6.3	27.4	28.1	77.1
4	D	3.2 (0.05)	84.4	3.9	29.0	29.2	82.3
5	E	5.6 (0.15)	84.2	4.9	30.6	31.0	80.9
6	F	5.6 (0.29)	82.4	4.4	31.1	31.4	82.0
7	G	4.5 (0.08)	80.4	5.3	32.4	32.8	80.7

[a] Keys for composition sample codes are in Table 1.
[b] Standard deviations in parentheses.

Table 3 lists HMF levels and colour parameters of roasted samples at 175 °C for 30 and 60 min, respectively. HMF level increased with roasting time, both in reference samples (nos. 8 and 14) and in defatted (nos. 9 and 15) or partially defatted samples (nos. 10–13, 17–20). In all cases, reference samples (8 and 14) showed the highest levels of HMF, increasing from 66.5 mg/kg in the sample roasted for 30 min to 144 mg/kg in the sample roasted for 60 min. In defatted or partially defatted samples, the HMF levels were almost the same, irrespective of the time of roasting. In fact, roasting for 30 and 60 min led to HMF levels of 17.9 and 19.9 mg/kg in defatted samples, respectively, while it led to 36.0 and 37.6 mg/kg in samples containing 40% of oil, respectively. HMF levels increased with the amount of oil, but were always far from the corresponding reference samples, as was evident in sample 21, which had the same percentage of oil as the reference sample (60%); in fact the level of HMF did not exceed 47 mg/kg. The effects of oil on HMF formation and colour parameters were very different when the oil was already contained in hazelnuts (samples 1, 8, 14) than when it was added (samples 4–7, 10–13, 17–21). In the latter it was less effective. Perren and Escher (1999) reported alteration of microstructure of hazelnuts, due to roasting, which induces a high mobility of the oil and of O_2 among the hazelnuts cells. The different oil distributions in hazelnuts, between the reference and the corresponding sample

Table 3
HMF concentration and colour parameters in roasted samples at 175 °C for 30 and 60 min

No.	Samples[a]	mg HMF/kg DCH[b]	L^*	a^*	b^*	C	h
175 °C for 30 min							
8	A	66.5 (5.62)	46.5	19.5	32.8	38.1	59.2
9	B	17.9 (0.66)	67.9	14.4	32.4	35.5	66.0
10	D	18.7 (1.43)	69.4	14.2	35.0	37.8	68.0
11	E	22.8 (0.04)	65.3	15.5	34.8	38.1	66.1
12	F	30.9 (1.67)	60.3	16.9	35.2	39.0	64.3
13	G	36.0 (1.46)	64.1	16.5	34.7	38.4	64.6
175 °C for 60 min							
Samples treated with oil							
14	A	14.4 (1.48)	35.8	22.5	26.1	34.4	49.2
15	B	19.9 (0.37)	58.4	18.4	31.2	36.9	60.0
16	C	16.3 (0.48)	52.0	18.4	30.2	35.4	58.6
17	D	32.5 (0.49)	54.9	19.2	33.1	39.0	60.9
18	E	34.3 (1.14)	52.7	18.9	33.0	39.0	60.8
19	F	36.7 (0.15)	54.7	18.9	33.9	40.9	60.9
20	G	37.6 (1.15)	50.7	20.6	34.2	39.3	59.4
21	H	47.1 (0.86)	49.7	19.9	33.1	38.6	59.0
22	I	33.3 (1.20)	53.4	19.8	33.9	39.2	59.7
Samples treated with oil containing hexanal							
23	L	33.6 (0.18)	55.5	18.7	32.1	37.2	59.8
24	M	39.0 (1.34)	56.7	18.8	34.0	38.9	61.0
25	N	34.7 (2.04)	56.2	18.5	33.0	37.9	60.7
26	O	36.7 (5.76)	52.2	20.1	33.4	39.0	59.0
27	P	45.0 (0.25)	50.4	20.6	33.1	38.9	58.1
28	Q	36.2 (1.31)	54.9	20.7	34.6	40.1	59.8
29	R	38.0 (1.59)	56.6	19.1	34.1	39.0	60.8
30	S	39.8 (2.68)	54.3	19.5	34.3	39.5	60.4
Samples treated with sucrose and oil containing hexanal							
31	J	372 (10.5)	29.6	19.9	21.2	29.1	46.8
32	K	33.5 (0.91)	59.1	17.3	30.6	35.2	60.5
33	T	51.5 (2.7)	53.5	19.4	33.4	38.6	59.8
34	U	49.5 (1.08)	56.2	17.6	32.0	36.5	61.2
35	V	99.5 (3.99)	52.0	19.7	32.7	38.2	58.9
36	W	108 (16.8)	55.5	17.6	32.3	36.8	61.4

[a] Keys for composition sample codes are in Table 1.
[b] Standard deviations in parentheses.

Table 4
R^2 values of linear correlation

Samples	L^*	a^*	b^*	C	H
HMF concentration/colour parameters					
1–7	−0.6254	0.6959	0.8874	0.8896	−0.4831
8–13	−0.9675	0.9649	−0.3479	0.3903	−0.9663
14–21	−0.9417	0.9171	−0.7428	−0.4977	−0.9381
Oil percentage/colour parameters					
4–7	−0.9552	0.7893	0.9828	0.9776	−0.6291
10–13	−0.7195	0.8867	−0.3341	0.6904	−0.9136
17–21	−0.8468	0.6169	0.1950	0.1745	−0.9002

treated with the same amount of oil (60%) could explain the reduced levels of HMF.

In order to assess whether, if in these systems, oil had only a solvent or a specific effect, a sample was prepared (no. 22) by adding (inert) paraffin oil (30%), instead of hazelnut oil. HMF concentration and colour parameters were very similar to the corresponding sample containing 30% of hazelnut oil (sample 19). Thus the oil has a solvent effect, confirming previous findings (Arnoldi & Corain, 1996).

Colour parameters show that an increase in roasting time led to an increase in brown colour products for all samples. Chiou, Tseng, and Ho (1991) found that L^* decreases with roasting time, while a^* and b^* increase. As well as HMF, colour parameters of reference samples (nos. 8 and 14) show a much greater extent of browning than defatted samples (nos. 9 and 15) or partially defatted samples (samples 10–13, 17–21). Also, in these systems, the reference samples (nos. 8 and 14) had the lowest L^* and h values, and the highest a^* values, b^* and C values were in the sample roasted for 30 min, not different from the other samples; instead, for samples roasted for 60 min, b^* and C values were the lowest. This indicated that the samples roasted for 60 min were the darkest.

There was no linear correlation between HMF concentration and oil percentage of samples, roasted at 150 °C, containing increasing amounts of oil (samples 4–7) ($R^2 = 0.1951$), and between HMF concentration and colour parameters (Table 4; samples 1–7), probably because the reaction of HMF formation was at the beginning, as indicated by its low concentration in these samples. On the other hand the colour parameters L, b^* and C seems to be well correlated with the oil percentage in samples 4–7 (Table 4).

In samples roasted at 175 °C for 30 and 60 min (samples 10–13, 17–21), HMF level was very highly correlated with the amount of oil ($R^2 = 0.9860$; $R^2 = 0.9326$, respectively), while only some colour parameters, such as L^*, a^* and h, were well correlated with the HMF concentration (samples 8–13, 14–21) (Table 4). Table 5 lists the R^2 of linear correlation between oil percentage and colour parameters; only the hue parameter seemed to be correlated with the oil percentage, in samples 10–13 and 17–21 (Table 4).

Hazelnuts cv *Mortarella*, had a 63.4% oil content, with 74% oleic acid and 16% linoleic acid (Ebrahem, Richardson, Tetley, & Mehlenbacher, 1994). Degradation of these acids leads to the formation of off-flavours, such as octanal and hexanal, respectively (Forss 1972; Frankel 1982, 1984). Kinderler and Johnson (1992) found that the oxidation of linoleic acid occurred more readily than oleic acid, as confirmed by the highest concentration of hexanal in hazelnuts stored for a long time at room temperature.

In order to evaluate the effect of hexanal on HMF and colour development during roasting, samples of DCH were added to oil containing different amounts of hexanal (Table 1). In particular, two series of samples were prepared treated with 10% (samples 23–25) and 30% of oil (samples 26–30), containing hexanal

(Table 3). The same samples, roasted at 150 °C, did not show any differences from the corresponding samples without hexanal (data not shown). In samples roasted at 175 °C, the addition of 10 or 30% of oil, containing hexanal, led to a small increase of HMF, only in samples with the highest concentrations of hexanal (samples 24–25 and 29–30). Colour parameters of all samples did not show significant differences from samples containing 10 or 30% of hazelnut oil (samples 17 and 19).

Frequently the food industry cooks hazelnuts in the presence of sucrose. For this reason, a set of experiments using sucrose were carried out. In samples 31 and 32, sucrose was added to the reference sample and to the DCH, respectively. After roasting, HMF reached the highest value in sample 31 (372.4 mg/kg), and a very low value in defatted sample 32 (33.5 mg/kg), confirming the importance of oil during the heating process. Both values were higher than in the corresponding samples without sucrose (samples 14 and 15). Colour parameters showed that sample 31 was more brown than sample 14, while sample 32 was very similar to sample 15. When hazelnut oil was added to the DCH/sucrose system (samples 33), roasting led to the HMF level of 51.5 mg/kg. A similar value (50.3 mg/kg) would be obtained by adding to the HMF formed in sample 15 (19.9 mg/kg) the differences due to the addition of sucrose (samples 24 minus 15, 13.6 mg/kg) and to the addition of 30% of oil (samples 19 minus 15, 16.8 mg/kg). In the same system treated with increasing amounts of hexanal (samples 34–36), roasting led to higher HMF levels, up to 99.5 and 108 mg/kg (samples 35 and 36, respectively) than the corresponding sample without hexanal (sample 33). Notwithstanding the increase of HMF concentration, colour parameters of these samples were very similar to those of sample 33, indicating that the addition of oil containing hexanal induced much more formation of HMF, while colour seemed not to be influenced, confirming previous findings obtained in model systems (Fallico & Ames, 1999).

These results show that the presence of oil, hexanal and sucrose, as well as increases of time and temperature, during hazelnut roasting, are very important for colour development and for the final HMF level. The role of oil during roasting seems to be independent of its chemical composition, but it seems to be related to the presence of lipid oxidation products, for example, aldehydes. This effect is evident at very high temperature. Recently, some authors have proposed, in order to achieve low-calories and low-fat products, use of defatted hazelnut flour. Results reported in this paper show that the extraction of oil from hazelnuts gives crushed hazelnuts with characteristics, during roasting, completely different from the starting material. Furthermore, partial or total addition of oil to DCH does not allow the same colour development, browning, and HMF levels of crushed hazelnut.

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