HEATING ON THE VOLATILE COMPOSITION AND SENSORY ASPECTS OF EXTRA-VIRGIN OLIVE OIL

Aquecimento na composição volátil e aspectos sensoriais do azeite de oliva extra virgem

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ABSTRACT

The main ways by which extra-virgin olive oil is consumed include direct application on salads or as an ingredient in sauces, but it is also been used by some for cooking, including frying and baking. However, it has been reported that under heat stress, some nonglyceridic components of olive oil are degraded. So, the effect of heating (at 50, 100, 150, and 200 °C for 2 h) on the volatile composition and sensory aspects of extra-virgin olive oil were evaluated. Heating altered the volatile composition of extra-virgin olive oil, mainly at higher temperatures (above 150 °C). The main modifications were related to the formation of large amounts of oxidized compounds, particularly large chain aldehydes. Sensory aspects were also altered when the oil was heated to higher temperatures, which might have occurred because of color alterations and mainly changes in the volatile composition of the oil.

Index terms: Vegetable oil, sensory analysis, chemical composition.

RESUMO

Os principais meios de consumo do azeite de oliva extravirgem incluem a aplicação direta em saladas ou como ingrediente em molhos, mas ele também tem sido usado para cozinhar, seja em frituras ou cozimentos. No entanto, sob estresse térmico, alguns componentes não-glicerídicos do azeite podem ser degradados. Assim, o efeito do aquecimento (a 50, 100, 150 e 200 °C por 2 h) sobre os componentes voláteis e aspectos sensoriais do azeite de oliva extravirgem foram avaliados. Os resultados indicaram que o aquecimento alterou a composição volátil do azeite extra virgem, principalmente em temperaturas mais elevadas (acima de 150 °C). As principais modificações foram relacionadas à formação de compostos oxidados, sobretudo aldeídos de cadeia longa. Os aspectos sensoriais também sofreram alterações quando o azeite foi aquecido a temperaturas elevadas (acima de 150 °C), o que pode ter sido influenciado por alterações de cor e pelas da composição volátil do produto.

Termos para indexação: Óleo vegetal, análise sensorial, composição química.

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INTRODUCTION

Extra-virgin olive oil is a product appreciated for its flavor, which is distinguished from that of other vegetable oils, and is used in various culinary practices worldwide. Extra-virgin olive oil is obtained from the first cold pressing of olives, which produces a special volatile compound composition that provides its much-appreciated characteristics of taste and aroma (OLIVEIRA et al., 2008).

Since no refining process is employed, extra-virgin olive oil contains a number of nonglyceridic constituents, which are believed to account for its beneficial health effects due to their considerable antioxidant activity (VISIOLI et al., 2006). In addition, extra-virgin olive oil has been reported to have anticancer, anti-inflammatory, and antiatherogenic activities (BERGER; JONAS; ABUMWEIS, 2004). Furthermore, its high monounsaturated fatty acid composition has been associated with the prevention of some pathologies, particularly cardiovascular diseases (MESA et al., 2006). Therefore, the consumption of extra-virgin olive oil has been increasing and research has been encouraged aimed at improving the production (DUTRA et al., 2004; OLIVEIRA et al., 2009; OLIVEIRA et al., 2010).

Although the main ways by which extra-virgin olive oil is consumed include direct application on salads or as an ingredient in sauces, it is also been used by some for cooking, including frying and baking. However, it has been reported that under heat stress, some nonglyceridic components of olive oil are degraded, such as chlorophylls, carotenoids (AVADI; GRATI-KAMOUN; ATTIA, 2009), tocopherols (ALLOUCHE et al., 2007), and especially phenolic compounds (CERRETANI et al., 2009; BENDINI et al., 2009; VALLI et al., 2010;). In addition, parameters related to the hydrolytic and oxidative states of the oil also are altered under heat stress (CERRETANI et al., 2009; BENDINI et al., 2009).

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Triglycerides can also degrade under thermal stress, thus affecting the quality of the oil. Studies on soybean oil and soybean oil-based fats showed that volatile compounds (SNYDER; KING, 1994) and sensory characteristics (BREWER et al., 1999) were altered after intense heating. The main alterations in volatile composition are the formation of oxidized compounds, particularly specific aldehydes, which were negatively correlated with sensory aspects.

This work evaluates the effect of heating on the volatile composition and sensory aspects of extra-virgin olive oil. Samples of extra-virgin olive oil were heated at 50 °C, 100 °C, 150 °C, and 200 °C for 2 h.

MATERIAL AND METHODS

Sample preparation

Samples of extra-virgin olive oil were obtained from a local market in Lavras, Minas Gerais, Brazil. The samples (from the same mark and lot) were sold in 500 mL amber glass bottles. No test has been carried out to attest the authenticity of product.

For the heat treatments, 250 mL samples of extra-virgin olive oil were put in 1,000 mL beakers and heated in a temperature-controlled electric furnace (Layr model Jady) for 2 h. The samples were heated at 50 °C, 100 °C, 150 °C, and 200 °C. An unheated reference sample also was prepared. After the heat treatment, the samples were protected from light until analysis (after 24 h).

Sensory analysis

The effect of heating on the sensory aspects of extra-virgin olive oil was evaluated by a difference from the control test. This test was used to determine if there was, in fact, a difference between the test samples and the control, and if there was a difference, its size was also measured (MEILGAARD; CIVILLE; CARR, 2007; LAWLESS; HEYMANN, 2010).

First, a training test was given to regular consumers of extra-virgin olive oil. This type of tester was used in order to reproduce the effect of heating on the perception of common consumers of extra-virgin olive oil. So, expert olive oil tasters were not employed. For the experiments, the testers were asked to discriminate 3 samples (100% olive oil, and 80% and 60% olive oil blended with soybean oil) from a control (100% olive oil) based on flavor and overall aspects using the following scale: 0 - no difference, 1 - very small difference, 2 - small difference, 3 - small/moderate difference, 4 - moderate difference, 5 - moderate/large difference, 6 - large difference, and 7 - very large difference. The experiments were repeated in quadruplicate. The more capable (those with better repeatability and discriminative ability) testers were selected for the experiments.

The 10 selected trained testers were then asked to discriminate samples of extra-virgin olive oil heated at 50 °C, 100 °C, 150 °C, and 200 °C from a unheated control based on flavor and overall aspects using the scale described above. The evaluations were carried out in triplicate. The results were expressed as the means of the difference from the control given by the testers.

Experiments were conducted in a closed cabin with white illumination. The samples (5 mL of extra-virgin olive oil) were labeled with 3 random digits on a white surface.

Color analysis

Color parameters of the extra-virgin olive oil samples were measured in triplicate using a Minolta CR-400 colorimeter based on the CIE-L*a*b* color system.

Volatile compound analysis

A headspace was created by heating 5 mL of extra-virgin olive oil at 40 °C for 15 min in a 10.0 mL glass flask. The compounds in the vapor phase were extracted by solid phase microextraction using a PDMS/DVD fiber. After 15 min, the fiber was injected in a gas chromatograph/mass spectrometer (GCMS-QP2010 plus; Shimadzu, Kyoto, Japan). The injection was made in splitless mode with helium as carrier gas at a flow rate of 1.0 mL min^{-1}. The injector temperature was 220 °C. A fused-silica capillary column (5% phenyl-95% polydimethylsiloxane, 30 m × 0.25 mm, 0.25 μm) was employed for separation of the compounds. The oven temperature was programmed from 35 °C (for 2 min) to 250 °C at a rate of 5 °C min^{-1}, and was then maintained at 250 °C for 1 min. The mass spectrometer was used with electron ionization (70 eV) and a mass scan range from 40 to 600 Da. The temperatures of the ion source and the GC-MS interface were 200 °C and 230 °C, respectively. Compounds were identified by comparing their mass spectra with GC/MS spectral libraries (Wiley 8 and FFNSC 1.2 libraries). A comparison of relative retention index (obtained using a series of n-alkanes under the same operational conditions) with references from databases (ADAMS, 2007; EL-SAYED, 2012; ACREE; ARN, 2012) was also used for compound identification. The relative abundance of the compounds was expressed as the relative chromatographic peak area (normalized area, %).

Statistical analysis

The data from the sensory tests were analyzed by Dunnett's test at the 5% significance level. The data from

the color characterization were analyzed by Tukey's test at the 5% significance level. All calculations were performed using SensoMaker software (PINHEIRO; NUNES; VIETORIS, 2013).

The volatile composition data was explored using principal component analysis (PCA). A m × n matrix, where m is the number of samples and n is the number of volatile compounds was constructed using the relative areas of each chromatographic peak identified. The data were autoscaled and PCA was run. The PCA routines were performed using Chemoface software (NUNES et al., 2012).

RESULTS AND DISCUSSION

Volatile compound profile

The flavor of extra-virgin olive oil is produced by a variety of compounds, such as aldehydes, alcohols, carboxylic acids, and esters, among others (KALUA et al., 2007). Different classes of volatile compounds were identified in the heated and unheated extra-virgin olive oil (Table 1). Olive oils that were unheated or heated at a lower temperature (50 °C) had higher alcohol, ester, and terpene content than oils heated at higher temperatures, which had higher total aldehydes and carboxylic acids.

Table 1 – Volatile compounds identified by SPME-GC/MS in extra-virgin olive oil heated at different temperatures (no heat, 50, 100, 150 and 200 °C).

N°	compound	RT	main m/z	RI	relative peck area (%)				
					no heat	50 °C	100 °C	150 °C	200 °C
1	3-methylbutanol	4.74	55,70,57	747	0.88	0.59	0.28	0.00	0.00
2	1-pentanol	5.51	55,57,70	775	0.14	0.31	0.33	0.43	0.75
3	Z-2-pentenol	5.61	57,55,68	778	0.31	0.53	0.17	0.75	0.04
4	Hexanal	6.33	56,57,72	801	2.53	2.54	2.65	2.59	4.32
5	Ethyl butyrate	6.43	71,88,60	805	1.10	1.86	0.63	0.55	2.34
6	E-2-hexenal	7.9	55,69,83	858	12.0	9.78	6.26	0.41	0.07
7	Z-3-hexenol	8	67,55,82	861	9.59	7.85	6.35	0.24	0.00
8	Z-2-hexenol	8.33	57,82,67	871	4.09	3.77	2.37	0.08	0.00
9	Hexanol	8.42	56,55,69	874	4.91	3.94	2.50	0.24	0.15
10	3-methylbutyl acetate	8.62	55,70,63	880	0.72	0.44	0.11	0.00	0.00
11	Heptanal	9.43	72,57,84	904	1.65	0.53	1.76	2.48	2.64
12	E-2-heptenal	11.2	55,83,70	961	1.52	1.69	3.76	3.86	3.09
13	Myrcene	12.3	81,58,69	992	1.76	0.11	0.12	0.05	0.04
14	E,Z-2,4-heptadienal	12.5	81,53,110	998	2.69	2.38	5.28	2.34	0.87
15	Octanal	12.7	56,84,69	1004	0.89	1.22	1.12	4.71	4.77
16	Z-3-hexenyl acetate	12.8	67,82	1009	8.48	8.83	6.48	0.23	0.49
17	Hexyl ethanoate	13	56,61,69	1015	1.13	0.95	0.49	1.45	1.87
18	E-beta-ocimene	14.1	93,79,105	1052	2.03	1.87	1.20	0.16	0.09
19	E-2-octenal	14.5	55,70,83	1063	2.42	1.28	1.21	2.29	2.07
20	Octanol	14.8	56,69,84	1074	0.57	0.61	1.23	1.07	1.25
21	Nonanal	15.9	57,70,82	1106	3.86	7.00	13.5	16.4	16.5
22	2-ethyl hexanoic acid	16.4	73,88,57	1123	0.00	0.20	0.37	0.51	0.18
23	E-2-nonenal	17.6	55,70,83	1164	0.29	0.76	1.01	2.58	2.51
24	Octanoic acid	18	60,73,101	1177	0.47	0.74	2.22	3.16	1.90
25	Decanal	18.9	55,69,97	1206	27.4	18.4	13.8	2.65	1.14
26	E-2-decenal	20.6	70,55,83	1265	3.73	9.29	9.16	17.1	20.1
27	Nonanoic acid	21.1	60,57,73	1280	0.05	1.16	4.02	9.26	6.13
28	E,E-2,4-decadienal	22.2	81,67,55	1319	3.08	5.91	4.53	7.17	5.97
29	E-2-undecenal	23.5	70,57,83	1366	1.75	5.44	7.02	17.3	20.7

RT: retention time; RI: retention relative index.

A decrease in the alcohols, esters, and terpenes was observed with increased heating temperature, whereas an increase in oxidized compounds, such as aldehydes and carboxylic acids, was observed with increased heating temperature (Figure 1), suggesting more drastic deterioration of olive oil at higher temperatures. In fact, high rates of aldehyde formation (mainly nonanal), in addition to increased carboxylic acids, have been observed during the oxidation of olive oil (KIRITSAKIS, 1998). These compounds are reported to negatively influence the sensory characteristics of oils (VICHI et al., 2003; KALUA et al., 2007).

An exploratory analysis of the volatile profile of the heated and unheated olive oils was performed by PCA. A tendency to differentiate heated samples from unheated samples was observed along the PC1 score axis (Figure 2-A), which explains approximately 70% of the variance. This separation of the samples along PC1 increased with higher heating temperatures. Therefore, the volatile profile of olive oil heated at 50 °C was more similar to that of the unheated sample, while the olive oils heated at 150 °C and 200 °C were more different than that of the unheated sample. The sample heated at 100 °C differed slightly from that of the unheated olive oil.

The loadings plot revealed alterations in the abundance of volatile compounds according to heat treatment (Figure 2-B). In general, alcohols and aldehydes with 6 carbons were abundant in unheated and mildly heated olive oils, whereas carboxylic acids and long chain saturated and unsaturated aldehydes were abundant in oils heated at high temperatures. Alcohols, including 3-methylbutanol, Z-3-hexenol, Z-2-hexenol, and hexanol, in addition to E-2-hexenal, decanal, 3-methylbutyl acetate, Z-3-hexenyl acetate, E-beta-ocimene, and myrcene, were more abundant in olive oils that were unheated and heated at 50 °C. Aldehydes, including heptanal, octanal, nonanal, E-2-nonenal, E-2-decenal, E,E-2,4-decadienal, and E-2-undecenal, in addition to 1-pentanol, octanol, and nonanoic acid were higher in olive oils heated at 150 °C and 200 °C. In addition, E-2-heptenal, 2-ethyl hexanoic acid, and octanoic acid were more abundant in olive oil heated at 150 °C; hexanal and hexyl ethanoate were more abundant in olive oil heated at 200 °C.

Some compounds abundant in heated oils, such as heptanal, nonanal, E-2-decenal, and E-2-undecenal, are aldehydes formed by the thermal degradation of oleic acid during the heating process, while the aldehydes hexanal, E,E-2,4-decadienal, E-2-heptenal, and E-2-octenal are formed by the decomposition of linoleic acid. Hexanal and nonanal are widely used to assess lipid oxidation (KIRITSAKIS, 1998; KALUA et al., 2007).

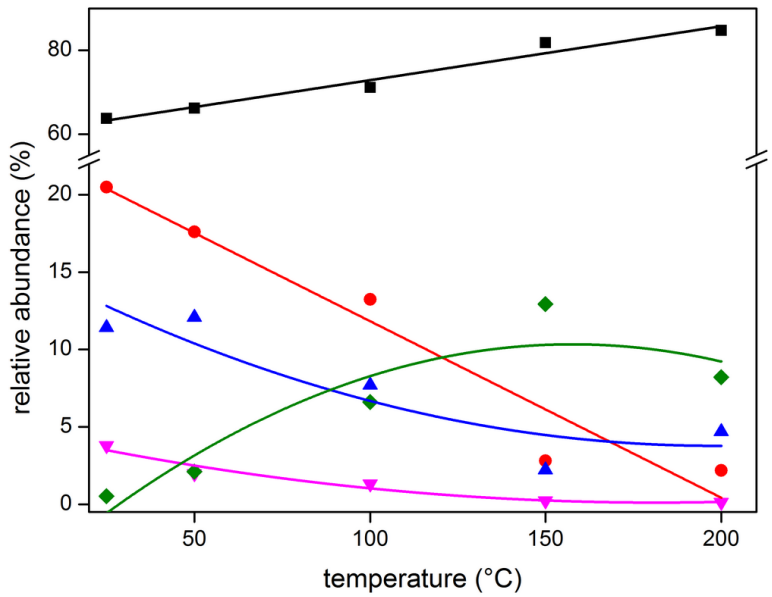

Figure 1 – Relative abundance of aldehydes (■), alcohols (●), esters (▲), terpenes (▼), and carboxylic acids (♦) in extra-virgin olive oil heated at different temperatures. Their relative abundances were obtained by the sum of the respective relative peak areas given in table 1.

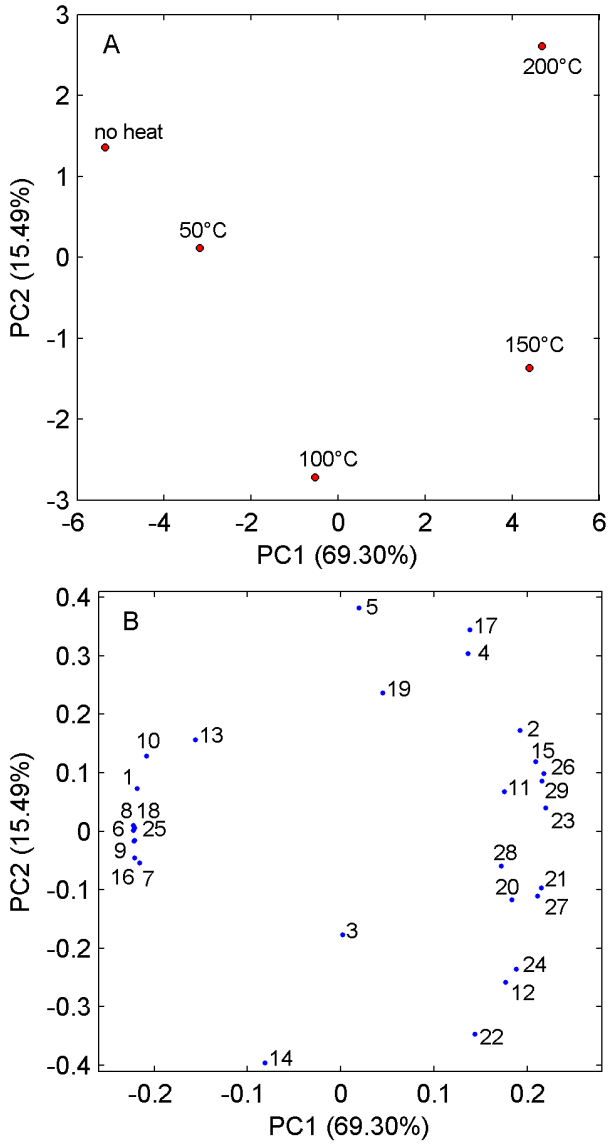

Figure 2 – Principal component analysis for the volatile compound profiles of extra-virgin olive oil heated at different temperatures. The loading points were numbered according to the compounds shown in table 1.

The volatile profiles are directly linked to the sensory perception of olive oil. Aldehydes and alcohols with 6 carbon atoms are associated with sweetness and contribute favorably to the aroma of extra-virgin olive oil. Esters are predominantly linked to positive fruity aroma. In contrast, carboxylic acids are linked to sour and pungent sensations and are associated with sensory defects in olive oil, mainly rancidity (APARICIO; MORALES; ALOSO, 1996; KALUA et al., 2007).

Sensory evaluation

The effect of heating on the sensory aspects of extra-virgin olive oil was evaluated by a difference from the control test. The flavor and overall aspects of the heated samples were compared to those of an unheated sample (control). The means of the differences from the control perceived by the testers are presented in table 2. The difference from control increased as heating temperature increased. However, only extra-virgin olive oils heated at 150 °C and 200 °C differed significantly from the unheated olive oil (control), corroborating the difference observed in the PCA of the volatile composition (Figure 2). According to the difference scores, the flavor of the oil heated at 150 °C had a moderate/large difference compared to that of the unheated control (difference scores of 4–5), whereas the overall aspects of oil heated at 200 °C had a large difference (difference score of approximately 6).

Table 2 – Difference from the control tests for extra-virgin olive oil heated at different temperatures.

	flavor		overall aspect	
	difference from control	p*	difference from control	p*
no heat	1.29	-	1.46	-
50°C	1.67	0.724	1.25	0.963
100°C	2.21	0.060	1.88	0.703
150°C	4.71	0.000	4.17	0.000
200°C	6.42	0.000	5.67	0.000

* p-value for Dunnett test at 5% significance.

The color alteration observed in the olive oil heated at 200 °C can, in part, explain the larger difference from the control; olive oil heated at 200 °C had less yellowish/greenish coloration than is typical of extra-virgin olive oil. This was confirmed by instrumental color determination (Table 3). A significant difference in b* was observed for the olive oil heated at 200 °C, which was less yellowish (lower b*) than the other samples. The L* parameter also indicated a slight bleaching (higher L*) of the oils heated at 150 °C and 200 °C. Significant differences were not observed in the a* parameter, which indicates other color alterations. Degradation of the oil pigments at high temperatures, mainly chlorophylls, could explain these observed color changes (AYADI; GRATI-KAMOWN; ALONSO, 2009).

Table 3 – Color parameters of the extra-virgin olive oils heated at different temperatures.

Treatment	L*	a*	b*
no heat	30.58±0,19[a]	-0.93±0,03[a]	7.00±0,52[a]
50°C	30.97±0,20[a]	-0.87±0,10[a]	7.47±1,06[a]
100°C	30.85±0,28[a]	-0.89±0,16[a]	7.75±1,04[a]
150°C	31.08±0,06[ab]	-1.05±0,02[a]	6.16±0,29 [a]
200°C	31.50±0,11[b]	-0.98±0,01[a]	2.34±0,23[b]

Means with different letters on vertical indicates significant difference in Tukey test at 5% significance level.

The sensory differences in the olive oils heated at high temperatures (150 °C and 200 °C) could also be explained by the higher unsaturated C7–C12 aldehyde and carboxylic acid contents in these samples, since these have been reported as the main compounds responsible for the undesirable sensory characteristics of oxidized vegetable oils (KALUA et al., 2007). In contrast, the C6 aldehydes and alcohols, predominant in the unheated and mildly heated oils, contribute favorably to the aroma of extra-virgin olive oil (KALUA et al., 2007).

CONCLUSIONS

Heating of extra-virgin olive oil at lower temperatures (up to approximately 100 °C) does not considerably alter the volatile constituents; however, heating at higher temperatures (above 150 °C) causes significant alterations in the volatile fraction. The main modifications are related to the formation of large amounts of oxidized compounds, particularly saturated and unsaturated long chain aldehydes.

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