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Short communication

Solid-phase microextraction and gas chromatography–mass spectrometry of volatile compounds from avocado puree after microwave processing

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Abstract

Microwave processing offers an alternative to blanch fruits and vegetables, since the application of high temperature and short time often results in minimum damage. An experimental design was used to investigate the effect of microwave time, pH, and avocado leaves (independent variables) on avocado flavor (response) using solid-phase microextraction (SPME)–GC–MS. Among the fully characterized flavor volatiles, 19 compounds were derived from lipid oxidation and only 4 from the avocado leaves. The main components derived from lipids were aldehydes, ketones and alcohols. Terpenoids, estragole, and 2-hexenal [*E*] were volatiles derived from avocado leaves. When leaves were added to fresh and microwaved avocado terpenoids and 2-hexenal [*E*]/hexanal ratio increased, this behavior was considered to have a positive effect on the sensorial quality of the product. From the statistical analysis of the experimental design, it was possible to determinate that the most important factors influencing the abundance of flavor compounds derived from lipids were microwave time and pH. Maximum values of these compounds were detected at high levels of microwave time and low values of pH. On the other hand, response surface of terpenoids and estragole showed an increment when microwave time and avocado leaf was increased. The region of optimum response was 30 s microwave time, pH 5.5, and 1% of avocado leaf.

Keywords: Food analysis; Avocado; Response surface; Volatile organic compounds

1. Introduction

Several attempts have been made to process avocados as puree or "guacamole". However, products obtained with these methods show color and flavor deterioration [1]. Microwave processing offers an alternative way to blanch fruits and vegetables, since the application of high temperature and short time often results in minimum damage. Besides, previous studies have shown that better retention of color and lightness is obtained when avocado puree is processed in a microwave oven for 30 s with copper or zinc chloride addition at pH 5.5. This was mainly attributed to the formation of complexes of chlorophyll with copper and zinc and consequently enzymatic browning inhibition [2]. Solid-phase microextraction (SPME) is a solvent-free, simple, inexpensive, rapid, and versatile method for the extraction of volatile compounds [3]. In an attempt to fully characterize avocado puree flavor, a fiber of medium polarity (for flavors) was used to reduce discrimination toward very polar and very non-polar volatiles [4].

Our aims were to evaluate the volatile profiles of avocado purees after microwave treatment at different pH value and after the addition of avocado leaves by response surface analysis, and analysis by SPME–GC–MS.

2. Methodology

2.1. Response surface methodology

Response surface was used to investigate the effect of heating time (s), pH, and avocado leaf content (independent

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variables) on the volatile compounds' response of avocado puree expressed as absolute peak area. Microwave time was varied from 0 to 60 s and the amount of avocado leaves from 0 to 2%. On the other hand, the pH was varied from 4.5 to 6.5. A total of 20 runs, based on a central composite were performed for the study of these experimental factors. The statistical data analysis and response surface plots were done using a Design Expert statistical package. The dependent variable was the absolute area of volatile compounds. This design was done three times.

2.2. Sample preparation

Avocado puree (20 g) was placed on petri dishes (5 cm diameter × 1 cm height) and heated in a microwave oven Panasonic, 2450 MHz, 633 W, as calculated according to [5]. The energy (E) for the microwaves was calculated using the equation: $E = Wt/m$, where W is the microwave oven power (633 W), t is the time of microwave exposure in seconds, and m is the quantify of the sample (in g) [6]. In addition, the energy density on avocado puree heated at 30 and 60 s were 0.94 and 1.89 kJ/g, respectively. Previous reports mentioned that avocado oil extracted with microwaved treatment, the energy density in the 1.50–1.89 kJ/g is the one that presents the lowest levels of trans fatty acids (<0.5 g/100 g) [7].

2.3. SPME analysis

Volatiles were collected from 5 g of the avocado puree or 0.2 g of the avocado leaves for 30 min on a divinylbenzene–Carboxen–polydimethylsiloxane (DVB–CAR–PDMS) fiber in a 10 ml vial after equilibration for 24 h at room temperature. Fibers were reconditioned before each extraction by heating them at 220 °C in the GC injector port for 2 h. Volatiles were analyzed using a HP model 5890 gas chromatograph equipped with a HP-FFAP column (30 m × 0.25 mm i.d., film thickness 0.25 μm; Hewlett-Packard, Palo Alto, CA, USA) interfaced to a mass-selective detector (HP model 5985). The oven temperature was initially held at 40 °C for 5 min and then increased by 5 °C/min to 120 °C with a final hold time of 3 min. The injector and detector temperatures were 180 and 230 °C, respectively. Helium carrier gas was used. Mass spectra were recorded in the electron impact mode at 70 eV. Retention indices were calculated using an n-alkanes (C_7–C_{22}) series. Most compounds were identified by comparing their mass spectra with those of authentic compounds and also with computerized spectral databases (NIST/EPA/NIH 75K), Kovats indices, and published literature.

3. Results and discussion

Among the fully characterized flavor volatiles, 19 compounds were derived from lipid oxidation and only 4 from the avocado leaves. In unprocessed avocado purée, six different volatile compounds were found: ethanol, 3-methylbutanol, acetic acid, 3-hydroxy-2-butanone, hexanol, and pentenol. In contrast, the compounds found in processed avocado were aldehydes, alcohols and ketones, being 2-heptenal [E] the most abundant of these compounds, followed by octanal, 1-octen-3-one, and 2-octenal [E]. On avocado leaves, the main volatile compounds were identified as estragol, terpenoids, and 2-hexenal. In particular, terpenoids and volatile compounds derived from lipid degradation showed an interesting pattern depending on the variety, rapeness, and extracting method. Previous studies reported 23 volatile compounds in fresh avocado [8], the large difference compare to the results in this work may be due to the avocado variety as well as the extraction method used. When avocado leaves were added to avocado purée processed with microwaves, terpenoids (α-pinene, β-pinene, eucalyptol), and estragole were found in addition to lipid volatile compounds. This enriches the aroma of microwaved purée. A decrease of hexanal content, together with an increase in the 2-hexenal [E] level was also found. This last compound imparts fresh and green flavor notes, which improve the sensorial quality of the purée. The levels of α-pinene, β-pinene, 2-hexenal [E], and estragole increased after microwave treatment of avocado leaves, whereas limonene, eucalyptol, and copaene showed a slight decrement. Estragole and 2-hexenal [E] showed increments of approximately 1.1- and 3-fold, respectively (Table 1). Yousif et al. [9] observed that estragole present in sweet basil showed an increment of approximately 1.5-fold when dried using vacuum microwave. From the statistical analysis of the experimental design, it was possible to determinate that the most important factors influencing the abundance of flavor compounds derived from lipids were microwave time and pH. Surface response analysis showed that as pH decreases and heating time increases, there is an increase in volatile compounds derived from lipids, and thus lipid volatiles are strongly influenced by time and pH. The volatile compound content derived from avocado leaves increases when leaf content and heating time increase. The best response was obtained with a heating time of 30 s, a pH 5.5, and a leaf content of 1%. Our results suggest a contribution of avocado leaves on the presence of α-pinene, β-pinene, and estragole in avocado purees (Fig. 1). The response surface of furfural showed larger increments at higher heating times and lower pH values. It is well known that Maillard reaction depends on temperature, water content, heating time, and pH. Also, Amadori rearrangement products are only pH dependent. At neutral or acidic pH the 1,2-enolization is favored, generating furfural [10]. In general, the response surfaces results indicate that maximum levels of volatiles derived from lipid oxidation are generated at longer heating time (60 s) and lower pH value (4.5). This suggests that the optimum conditions to prepare avocado puree are 30 s heating time, pH 5.5, and 1% of avocado leaves.

Table 1
Volatile compounds of fresh avocado, avocado leaves, and microwaved avocado puree with and with avocado leaves

I[a]	Compounds	Fresh avocado	Avocado leaves	Microwaved avocado	Microwaved avocado added with avocado leaves
913	Etanol	+	+	+	+
934	Pentanal			+	+
949	α-Pinene		+		+
960	1-Penten-3-one			+	+
1104	Hexanal			+	+
1108	β-Pinene		+		+
1160	β-Myrcene		+		
1191	Limonene		+		
1195	Heptanal			+	+
1203	Eucalyptol		+		+
1220	3-Methyl-butanol	+		+	+
1228	2-Hexenal [*E*]		+	+	+
1262	Pentanol	+		+	+
1291	3-Hydroxy-2-butanone	+			
1295	Octanal			+	+
1309	1-Octen-3-one			+	+
1328	2-Heptenal [*E*]			+	+
1363	Hexanol	+	+	+	+
1434	2-Octenal [*E*]			+	+
1460	1-Octen-3-ol			+	+
1463	Acetic acid	+	+	+	+
1471	Copaene		+		
1483	Furfural			+	+
1542	2-Nonenal [*E*]			+	+
1572	Octanol			+	+
1580	Caryophyllene		+		
1660	2-Decenal [*E*]			+	+
1691	Estragole		+		+

[a] Kovats retention index.

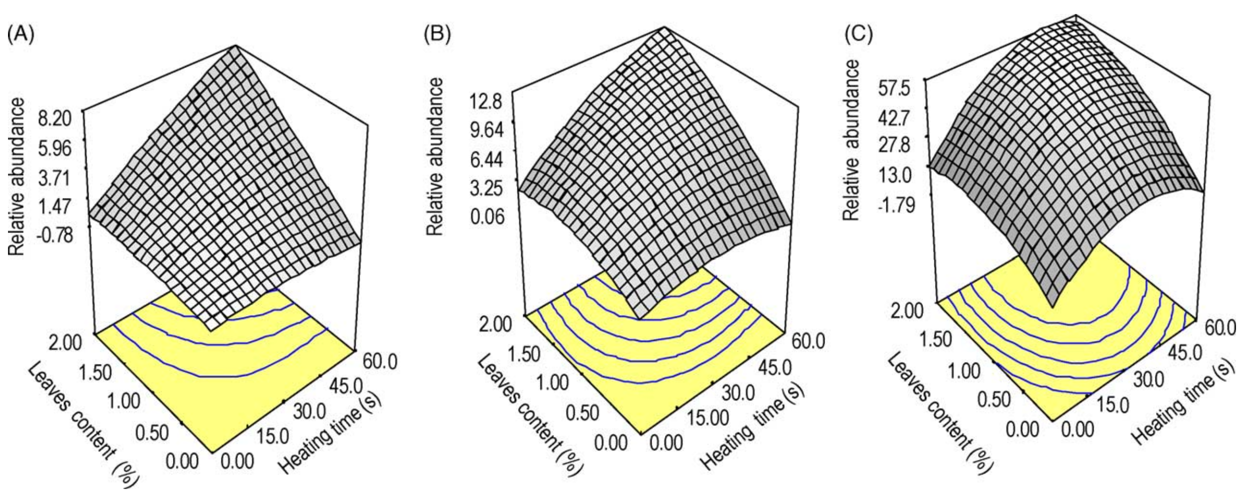

Fig. 1. Response surfaces of α-pinene (A), β-pinene (B), and estragole (C) plotted for heating time, avocado leaves addition, and fixed at pH 6.5.

4. Conclusions

SPME–GC–MS is a very useful tool to evaluate changes on avocado purees after a microwaved treatment. Main compounds were aldehydes, alcohols, ketones, and Maillard. However, larger amounts of estragole, terpenoids, and 2-hexenal [*E*] were found when avocado leaves were added to the purees. The response surface studies demonstrated the influence of heating time and pH on the generation of volatiles from lipid oxidation.

References

[1] A. López-Malo, E. Palou, G. Barbosa-Canovas, J. Welti-Chanes, B. Swanson, Food Res. Int. 31 (1998) 549.

[2] R. Guzmán, L. Dorantes, H. Hernández, H. Hernández, A. Ortiz, R. Mora, Inn. Food Sci. Emerg. Technol. 3 (2002) 47.
[3] C.L. Arthur, J. Pawliszyn, Anal. Chem. (1990) 2145–2149.
[4] P. Díaz, F.J. Señorans, G. Reglero, E. Ibáñez, J. Agric. Food Chem. 50 (2002) 6468.
[5] R. Buffler, Microwave Cooking and Processing; Engineering Fundamentals for the Food Scientist, AVI, New York, 1992, pp. 157–158.
[6] P.D. Rose, M.J. Connolly, Pharmacol. Ther. 83 (1999) 217.
[7] M.A. Ortiz, L. Dorantes, J. Galíndez, R.I. Guzmán, J. Agric. Food Chem. 51 (2003) 2216.
[8] S. Siyinda, J. Gramshaw, Food Chem. 4 (1998) 483.
[9] A. Yousif, C. Scaman, T. Durance, B. Girard, J. Agric. Food Chem. 47 (1999) 4777.
[10] C. Guyot, C. Bonnafotn, I. Lesschaeve, S. Issanchou, A. Voilley, E. Spinnler, J. Agric. Food Chem. 44 (1996) 2341.