

Volatile compounds present in six types of dry-cured ham from south European countries

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A study was conducted to compare the volatile fraction of six types of dry-cured hams from south European countries: (1) France: Bayonne and Corsican hams; (2) Spain: Iberian and Serrano hams; (3) Italy: Parma and Light Italian Country hams. The analysis of the volatile compounds was carried out using a dynamic-head space technique coupled to gas chromatography–mass spectrometry. One hundred and nine compounds were identified and analyzed: aldehydes (15), alcohols (14), ketones (17), esters (11), sulphur compounds (5), nitrogenous compounds (10), terpenes (18), *n*-alkanes (7) and aromatic and cyclic hydrocarbons (12). The possible formation pathways of these compounds are discussed. The results show that there are important differences among the six types of hams depending on the type of raw material and the technology used, the longer-processing time hams (Iberian and Corsican) being richer in volatile compounds. © 1998 Elsevier Science Ltd. All rights reserved

INTRODUCTION

Much attention has been devoted to the study of dry-cured ham in recent years (Toldra *et al.*, 1991; Palmia *et al.*, 1992; Buscailhon *et al.*, 1994; Elias *et al.*, 1994), especially to volatile compounds responsible for the flavour of dry-cured ham (Berdagué *et al.*, 1993). During the processing of dry-cured ham, there is a loss of water and a diffusion of salt throughout the ham, leading to a gradual stabilization of the product due to the drop of a_w . At the same time, there is a slow degradation of lipids and proteins, which produces an accumulation of free fatty acids and free amino acids, respectively (Flores *et al.*, 1988; López-Bote *et al.*, 1990). These compounds undergo further reactions to give volatile compounds (Berdagué and Garcia, 1990; Berdagué *et al.*, 1991, 1993; Garcia *et al.*, 1991; López *et al.*, 1992; Sabio *et al.*, 1995).

Different volatile compounds have been identified in dry-cured ham, e.g. carbonyl compounds (alkanals, alk-2-enals, alkadienals, and ketones), alcohols, short-chain fatty acids, sulphur compounds and nitrogenous compounds. The volatile compound composition of dry hams is markedly affected by the raw material and the technology; however, there has been no comparative

study between different types of hams. The aim of this paper is to compare the volatile fraction of six types of dry-cured ham from south European countries: (1) France: Bayonne and Corsican hams; (2) Spain: Iberian and Serrano hams; (3) Italy: Parma and Light Italian Country hams. This study is a part of a European project (AIR2.CT93.1757), which is attempting to establish a scientific basis for the control and improvement of the sensory quality of dry-cured hams.

MATERIALS AND METHODS

Ham processing

Two types of hams per country (France, Spain, and Italy) were processed by local manufacturers: Bayonne hams and Corsican hams in France, Iberian hams and Serrano hams in Spain, Parma hams and Light Italian Country hams in Italy. Thirty hams of each type were processed as follows.

Bayonne hams

The hams were rubbed with dry salt containing 0.5% added potassium nitrate, placed on platforms and held for 1 week at 4°C. Excess salt was removed, and hams were salted again and held for 1 week at 4°C. After

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washing to remove salt from the surface, they were left on platforms for 8 weeks, at 4°C and 65–85% relative humidity (RH). Then the hams were hung and heated at 20–25°C for 4 days and seasoned for 7 months at 12–15°C and 75–80% RH. The hams were made by two manufacturers following this scheme. One of the manufacturers rubbed the surface of the hams with pepper, and this group of hams is called Bayonne-2. The rest of the hams were not treated with pepper and called Bayonne-1.

Corsican hams

The fresh hams were put into salt for 4 days per kg fresh weight at 4°C, then washed and dried for several months before seasoning in a fresh cellar at 14–15°C and 75% RH for about 6 months. The total processing time was 18–24 months. In some cases, the ham surface was rubbed with black pepper.

Iberian hams

The raw material was from Iberian hams. The fresh hams were cut to remove subcutaneous fat (traditional V-shape). Then they were put into salt for 1 day per kg of weight, at 2–3°C. After salting, the post-salting period starts, which lasts 3 months. In this step, the hams were kept at a temperature below 10°C for 2 months and then the temperature was increased slowly up to 15–16°C. Then, the hams were put into a natural drying shed for 100 days, at 15–30°C and 60–80% RH. Finally, the hams were kept in a cellar for 12 months.

Serrano hams

The hams were rubbed with dry salt treated with potassium nitrate (0.3 g per kg of fresh ham) and held in salt for 10 days at 3°C. Salt in excess was removed by washing, and hams were kept at 5°C for 6 weeks. Then, they were placed in natural drying chambers at 12–16°C for 10 months.

Parma and Light Italian Country hams

Twenty-four hours after slaughter, the thighs were sprinkled with dry salt onto the muscular surface and wet salt was rubbed onto the skin. Placed on platforms, the hams were held for 1 week in a cooler; excess salt was then removed by brushing. The hams, re-salted, were kept for another 3 weeks in the same cooler. At the end of this step, excess salt was removed. Throughout the first and second salting, the temperature ranged from 0 to 4°C and the relative humidity from 70 to 90%. Then, the desalted hams were left to rest for three additional months in a dry cooler at 0–4°C and 60–85% RH. Ageing took 9 months for Parma hams and 5–6 months for Light Italian Country hams and was conducted at 15–17°C.

Sample preparation

Samples were taken from the Biceps Femoris muscle of the 180 hams. The samples were stored at –80°C, until

analysis. For volatile analysis, 20 g of frozen sample were minced with a home-mincer. Then, 5 g were weighed, with a precision of 0.01 g, in a flask for extraction of volatile compounds.

Isolation of volatile compounds

The isolation of volatile compounds was carried out with an Automatic Sampler Heater Tekmar 2016, controlled by a Purge and Trap Concentrator Tekmar 3000. The purge conditions were:

- Sample temperature: 35°C
- Tenax trap temperature: 35°C
- Purge flow: 100 ml min⁻¹
- Purge time: 1 h

After the purge time, the volatiles were desorbed and injected into the chromatographic column by holding the Tenax trap at 225°C for 1 min.

Chromatographic analysis

In all samples, the GC–MS analysis of the volatile compounds was performed by a Star 3400 Chromatograph (Varian) coupled to a Saturn 3 mass spectrometer (Varian). Mass spectra were acquired using a Compaq 4/50 computer, which also controlled the running conditions of the chromatographic analysis.

Gas chromatography was performed on a 50 m × 0.32 mm id fused silica DB5 column, coated with 5% diphenyl—and 95% dimethyl—polysiloxane of 1.0 μm film thickness, using helium as carrier gas. The column was held at 35°C for 5 min, and then the temperature was increased at 2°C min⁻¹ to 175°C. The mass spectrometer was scanned from m/z 10 to 400, under electronic ionization conditions. The ion source was held at 260°C. In some samples, chemical ionization was also carried out. In these cases, methane was used as chemical ionization reagent.

Statistical analysis

Mean and standard error were calculated for all quantified variables, except for aromatic hydrocarbons and alkanes because many of these compounds probably are contaminants from plastic wrapping and most likely have no significant impact on ham aroma (Stahnke, 1994, 1995). An analysis of variance (ANOVA) was carried out according to the model:

$$Y = \mu + T + r$$

where Y = volatile compound; μ = constant term; T = ham type; r = residual variation.

A principal component analysis of the most important volatile compounds was carried out. Finally, a stepwise discriminant analysis was done. All the statistical analyses were carried out using the SPSS programme.

RESULTS AND DISCUSSION

The results from the GC-MS (EI,CI) analyses are compiled in Table 1. Table 1 lists all the identified compounds. Table 2 shows the amount of those components and ANOVA results.

Identification

Table 1 shows the 109 volatile compounds which were identified and quantified in this study. These compounds can be clustered in the following chemical families: aldehydes (15), alcohols (14), ketones (17), esters (11), sulphur compounds (5), nitrogen compounds (10), terpenes (18), *n*-alkanes (7), and aromatic and cyclic hydrocarbons (12).

The identification was made after analyzing the MS-EI and MS-CI results, and comparing the Kovats indices obtained with those from the literature. So far as we know, 27 of these compounds were found for the first time in dry-cured hams, mainly terpenes, non-methyl ketones, and pyrazines.

ANOVA

Aldehydes

The results of the ANOVA analysis are shown in Table 2. Aldehydes constitute the most important family of volatile compounds from a quantitative point of view. These carbonyl compounds must play an important role in the aroma of the dry-cured ham because they have a low perception threshold.

All the aldehydes identified were present in the six types of hams, but Iberian hams had, by far, the highest value for most of these compounds. The exceptions were acetaldehyde, which is higher ($P < 0.05$) in Serrano, and benzaldehyde, which is higher in Corsican ($P < 0.05$).

Linear aldehydes, such as hexanal, heptanal, octanal, and nonanal, come mainly from an oxidative degradation of unsaturated fatty acids: oleic, linoleic, linolenic, and arachidonic (Frankel *et al.*, 1981; Chan and Coxon, 1987). On the other hand, the major formation pathway of the branched chain aldehydes seems to be the oxidative deamination-decarboxylation, probably via Strecker-degradation (Garcia *et al.*, 1991).

Alcohols

The origin of these compounds may be a chemical degradation or, perhaps, in part microbial activity may be involved. Their odour threshold value was higher than for aldehydes, so their influence in the aroma must be lower. However, unsaturated alcohols, such as 1-penten-3-ol and 1-octen-3-ol, each had a lower threshold value; thus these may play an important role in the odour (Seik *et al.*, 1977).

Unsaturated alcohols (1-penten-3-ol and 1-octen-3-ol) and pentanol concentrations were higher in Corsican

hams; branched alcohols (2-methylpropanol, 2 and 3-methylbutanol) and ethanol were abundant in Bayonne-2 and Serrano hams; secondary alcohols (2-pentanol and 2-heptanol) in Serrano hams; and medium-chain alcohols (hexanol and heptanol) in Iberian hams.

Ketones

Seventeen ketones were identified (Table 1), of which half are methyl ketones. The formation pathway of methyl ketones has been well documented because these compounds are responsible for the aroma of many blue cheeses (Creuly *et al.*, 1992). They have a very strong odour. In cheese, micro-organisms are involved in the formation of methyl ketones (Karahadian *et al.*, 1985). However, in normal hams, where the microbial population is relatively low, there are methyl ketones. It is very likely that these compounds are formed by a chemical process. Only in those cases where the amount of these compounds is abnormally high, should we think that micro-organisms may be involved.

The ANOVA results indicated that the Iberian hams were richer in medium chain length methyl ketones than the other groups.

Esters

These compounds have fruity notes, mainly those formed from short-chain acids. Esters with long-chain acids have a slight fatty odour. Esters are formed by esterification of carboxylic acids and alcohols (Mottram, 1991). In fact, Bayonne-2 and Serrano hams, which have a relatively high amount of ethanol, had the highest amount of ethyl esters, while Corsican hams had the highest amount of both 1-penten-3-ol and its acetate.

Sulphur compounds

These compounds come from the degradation of sulphur amino acids. They have a strong odour (Schutte, 1974). Corsican hams had the highest contents of dimethyl disulphide and dimethyl trisulphide, followed by Iberian. The high value of dimethyl disulphide in Corsican hams probably indicates the presence of some kind of alteration in some hams (Vidal-Aragón *et al.*, 1994).

Terpenes

The presence of limonene and other terpenes is usual in hams because these compounds are normal constituents of the unsaponifiable fraction of vegetable fat. Thus, they come from the feed and they are accumulated in the body of the animal (Buscailhon, 1992). However, the high amount of some terpenes found in Bayonne-2 and, to a lesser extent, in Corsican samples probably was due to the black pepper treatment on the surface of the ham during processing, because these compounds constitute 90% of pepper essential oil (Russell and Else, 1973). A further analysis of the volatile compounds of the surface of these hams verified the presence of a very

Table 1. Volatile compounds identified in Bayonna, Corsican, Iberian, Italian Country Style, Parma and Serrano hams

Compound	Method of identification	Compound	Method of identification
Aldehydes		Alcohols	
Acetaldehyde	EI/CI/KI	Ethanol	EI/CI/KI
2-Methyl propanal	EI/CI/KI	2-Methyl-3-buten-2-ol	EI/CI
3-Methyl butanal	EI/CI/KI	2-Methyl propanol	EI/CI/KI
2-Methyl butanal	EI/CI/KI	1-Penten-3-ol	EI/CI/KI
Pentanal	EI/CI/KI	2-Pentanol	EI/CI/KI
Hexanal	EI/CI/KI	3-Methyl butanol	EI/CI/KI
Heptanal	EI/CI/KI	2-Methyl butanol	EI/CI/KI
Benzaldehyde	EI/CI/KI	1-Pentanol	EI/CI/KI
2,4-Nonadienal	EI/CI/KI	1-Hexanol	EI/CI/KI
Octanal	EI/CI/KI	2-Heptanol	EI/CI/KI
2-Octenal	EI/CI/KI	Ethyl phenol	EI/CI/KI
Benzene acetaldehyde	EI/CI/KI	1-Heptanol	EI/CI/KI
Nonanal	EI/CI/KI	1-Octen-3-ol	EI/CI/KI
2-Nonenal	EI/CI/KI	Dodecanol	EI/CI
Decanal	EI/CI/KI		
Ketones		Esters	
2-Propanone	EI/CI/KI	Ethyl acetate	EI/CI/KI
2,3-Butanedione	EI/CI/KI	Ethyl propanoate	EI/CI/KI
2-Butanone	EI/CI/KI	Ethyl 2-methyl propanoate	EI/CI/KI
2-Pentanone	EI/CI/KI	Ethyl 2-methyl butanoate	EI/CI/KI
3-Hydroxy-2-butanone	EI/CI	Ethyl 3-methyl butanoate	EI/CI/KI
3-Penten-2-one	EI/CI/KI	1-Penten-3-ol acetate	EI/CI
3-Methyl-2-pentanone	EI/CI	Methyl hexanoate	EI/CI/KI
3-Hexanone	EI/CI/KI	Ethyl hexanoate	EI/CI/KI
2-Hexanone	EI/CI/KI	Pentyl butanoate	EI/CI/KI
Cyclohexanone	EI/CI/KI	Ethyl heptanoate	EI/CI/KI
4-Heptanone	EI/CI/KI	Ethyl octanoate	EI/CI/KI
2-Heptanone	EI/CI/KI		
4-Octen-3-one	EI/CI	Sulphur compounds	
6-Methyl-5-hepten-2-one	EI/CI	Carbon disulphide	EI/CI/KI
2-Octanone	EI/CI/KI	Dimethyl disulphide	EI/CI/KI
8-Nonen-2-one	EI/CI	Dimethyl trisulphide	EI/CI/KI
2-Nonanone	EI/CI/KI	Methyl <i>n</i> -pentyl disulphide	EI/CI
		Methyl <i>n</i> -hexane disulphide	EI/CI
Terpenes		Nitrogen-compounds	
Unknown terpene	EI/CI	<i>N</i> -Methylene ethanamine	EI/CI
Canphene	EI/CI	Pyrrrol	EI/CI
β -Phelandrene	EI/CI	<i>N,N</i> -Dimethyl metanamide	EI/CI
β -Pinene	EI/CI	Hexanonitrile	EI/CI
3-Carene	EI/CI	2,6-Dimethyl pyrazine	EI/CI
2,3-dihydrocineol	EI/CI	Trimethyl pyrazine	EI/CI
β -Myrcene	EI/CI	3-Ethyl-2,5-dimethyl pyrazine	EI/CI
α -Pinene	EI/CI	2-Ethyl-2,5-dimethyl pyrazine	EI/CI
Phelandrene	EI/CI	2,3-Diethyl-5-methyl pyrazine	EI/CI
2-Carene	EI/CI	3,5-Diethyl-2-methyl pyrazine	EI/CI
Unknown terpene	EI/CI		
Limonene	EI/CI	Aromatic and cyclic hydrocarbons	
Cineol	EI/CI	Methylcyclopentane	EI/CI
Unknown terpene	EI/CI	Toluene	EI/CI
Unknown terpene	EI/CI	Ethyl benzene	EI/CI
Unknown terpene	EI/CI	1,2-Dimethyl benzene	EI/CI
Unknown terpene	EI/CI	1,3-Dimethyl benzene	EI/CI
Caryophyllene	EI/CI	Ethyl, methyl benzene	EI/CI
		Trimethyl benzene	EI/CI
<i>n</i>-Alkanes		1-Ethyl-2-methylcyclopentane	EI/CI
Hexane	EI/CI	Trimethyl benzene	EI/CI
Heptane	EI/CI	Diethyl benzene	EI/CI
Octane	EI/CI	1,2,2-Trimethyl benzene	EI/CI
Nonane	EI/CI	Tetramethyl benzene	EI/CI
Undecane	EI/CI		
Dodecane	EI/CI		
Tridecane	EI/CI		

EI, Electron ionization; CI, Chemical ionization; KI, Kovats index.

Table 2. Mean amount (U. area/10000), standard error and ANOVA results of volatile compounds

Compound	Bayonne 1	Bayonne 2	Corsican	Iberian	LIC	Parma	Serrano
Aldehydes							
Acetaldehyde	3.8 ± 0.7 ^a	12.5 ± 1.9 ^a	4.8 ± 0.6 ^a	5.9 ± 0.6 ^a	8.1 ± 1.3 ^a	3.5 ± 0.4 ^a	27.0 ± 6.5 ^b
2-Methyl propanal	46.2 ± 5.2 ^a	124.2 ± 29.7 ^b	230.7 ± 17.3 ^c	210.9 ± 12.4 ^c	50.0 ± 3.7 ^a	46.7 ± 3.1 ^a	126.7 ± 9.3 ^b
3-Methyl butanal	888.8 ± 111.9 ^a	1249.9 ± 184.4 ^{ab}	2731.8 ± 216.3 ^c	4560.0 ± 483.6 ^d	1685.3 ± 169.4 ^{abc}	941.3 ± 87.3 ^a	2461.6 ± 200.4 ^{bc}
2-Methyl butanal	281.4 ± 39.4 ^a	399.5 ± 46.8 ^a	1794.0 ± 178.0 ^c	1982.0 ± 167.4 ^c	335.0 ± 30.1 ^a	278.8 ± 27.3 ^a	956.1 ± 78.2 ^b
Pentanal	185.2 ± 42.2 ^a	251.3 ± 53.4 ^{ab}	883.8 ± 171.3 ^b	2492.5 ± 239.9 ^c	369.1 ± 54.6 ^{ab}	225.2 ± 42.5 ^a	675.4 ± 114.9 ^{ab}
Hexanal	1103.1 ± 258.9 ^a	1516.1 ± 361.6 ^a	1744.5 ± 302.6 ^a	9571.2 ± 859.6 ^c	2057.0 ± 301.3 ^a	1486.3 ± 277.1 ^a	3195.4 ± 484.3 ^b
Heptanal	113.3 ± 22.4 ^a	113.0 ± 21.3 ^a	148.3 ± 23.1 ^a	888.5 ± 90.4 ^b	174.7 ± 25.5 ^a	128.8 ± 21.6 ^a	238.9 ± 35.4 ^a
Benzaldehyde	33.3 ± 7.58 ^a	55.7 ± 24.9 ^{ab}	89.4 ± 13.1 ^b	61.6 ± 8.4 ^{ab}	47.6 ± 7.7 ^a	34.5 ± 5.6 ^a	45.8 ± 5.6 ^a
2,4-Nonadienal	16.5 ± 4.1 ^a	22.2 ± 6.4 ^a	20.9 ± 4.7 ^a	70.8 ± 11.8 ^b	27.6 ± 3.7 ^a	23.1 ± 3.5 ^a	34.8 ± 4.0 ^a
Octanal	83.2 ± 16.3 ^a	75.4 ± 19.9 ^a	131.7 ± 18.4 ^a	249.7 ± 25.9 ^b	133.2 ± 18.3 ^a	120.0 ± 20.7 ^a	95.0 ± 16.5 ^a
2-Octenal	0.2 ± 0.1	0	0.8 ± 0.4	0.3 ± 0.2	0.4 ± 0.3	0.6 ± 0.5	0.9 ± 0.5
Phenylacetaldehyde	29.3 ± 15.7 ^a	211.7 ± 142.6 ^b	36.9 ± 4.1 ^a	123.2 ± 23.0 ^{ab}	47.8 ± 14.0 ^a	20.3 ± 5.8 ^a	35.3 ± 4.3 ^a
Nonanal	149.4 ± 26.3	134.2 ± 19.7	158.1 ± 22.1	195.4 ± 24.7	186.6 ± 22.4	172.4 ± 24.8	108.7 ± 14.5 ^a
2-Nonenal	1.4 ± 0.6	0.4 ± 0.5	1.7 ± 0.5	1.2 ± 0.3	1.3 ± 0.4	1.2 ± 0.3	0.9 ± 0.3
Decanal	6.4 ± 1.9	3.2 ± 1.5	9.9 ± 3.0	10.6 ± 2.2	8.0 ± 1.6	8.5 ± 3.2	4.0 ± 1.1
Alcohols							
Ethanol	120.4 ± 34.0 ^a	1747.6 ± 186.6 ^c	67.3 ± 12.6 ^a	121.0 ± 11.0 ^a	386.3 ± 66.5 ^{ab}	186.1 ± 24.6 ^a	935.8 ± 395.0 ^{bc}
2-Methyl-3-buten-2-ol	56.7 ± 5.7	46.9 ± 9.4	138.8 ± 26.0	73.4 ± 43.5	68.6 ± 8.3	66.8 ± 12.8	48.5 ± 20.5
2-Methyl propanol	29.6 ± 6.9 ^a	127.0 ± 21.7 ^c	99.9 ± 17.3 ^{bc}	35.5 ± 4.7 ^a	94.5 ± 17.6 ^{bc}	55.2 ± 10.2 ^{ab}	87.63 ± 13.2 ^{abc}
1-Penten-3-ol	107.7 ± 25.3 ^a	163.9 ± 25.6 ^a	1661.5 ± 178.6 ^b	261.5 ± 32.5 ^a	201.3 ± 25.5 ^a	133.6 ± 13.1 ^a	156.9 ± 16.6 ^a
2-Pentanol	5.7 ± 2.1 ^a	13.0 ± 3.1 ^a	76.7 ± 21.3 ^a	296.2 ± 38.4 ^b	10.6 ± 3.4 ^a	6.8 ± 2.2 ^a	303.1 ± 44.2 ^b
3-Methyl butanol	45.3 ± 11.9 ^a	574.2 ± 99.0 ^b	190.4 ± 68.2 ^a	163.1 ± 19.5 ^a	186.3 ± 77.0 ^a	80.0 ± 21.2 ^a	231.2 ± 35.0 ^b
2-Methyl butanol	0 ^a	221.9 ± 53.3 ^b	31.5 ± 16.8 ^a	1.7 ± 1.7 ^a	48.8 ± 37.5 ^a	8.3 ± 5.9 ^a	43.6 ± 14.1 ^a
1-Pentanol	100.2 ± 26.1 ^a	243.00 ± 52.1 ^a	814.7 ± 206.8 ^b	309.1 ± 31.8 ^a	268.5 ± 46.6 ^a	154.0 ± 30.1 ^a	271.8 ± 43.0 ^a
1-Hexanol	1.9 ± 1.9 ^a	21.1 ± 6.7 ^a	10.6 ± 3.5 ^a	285.8 ± 37.8 ^c	14.9 ± 4.4 ^a	8.5 ± 3.2 ^a	168.4 ± 29.4 ^{bc}
2-Heptanol	0 ^a	0 ^a	0.5 ± 0.2 ^a	21.0 ± 3.4 ^b	0 ^a	0 ^a	30.7 ± 5.4 ^b
Ethyl phenol	0.3 ± 0.2 ^a	0.2 ± 0.1 ^a	4.3 ± 1.0 ^{ab}	26.2 ± 2.7 ^c	0.2 ± 0.1 ^a	0.1 ± 0.1 ^a	7.1 ± 0.7 ^b
1-Heptenol	0.2 ± 0.1 ^a	0.8 ± 0.5 ^{ab}	3.2 ± 2.1 ^{ab}	4.9 ± 1.1 ^b	1.1 ± 0.7 ^{ab}	1.9 ± 0.9 ^{ab}	0.3 ± 0.2 ^a
1-octen-3-ol	30.7 ± 8.4 ^a	51.9 ± 13.4 ^a	106.4 ± 21.2 ^b	54.4 ± 6.0 ^a	66.3 ± 9.8 ^{ab}	53.7 ± 10.0 ^a	80.9 ± 10.2 ^{ab}
Dodecanol	1.1 ± 0.9 ^{ab}	0.5 ± 0.3 ^a	1.2 ± 0.9 ^{ab}	5.3 ± 2.5 ^b	1.1 ± 0.7 ^{ab}	1.3 ± 1.0 ^{ab}	0.2 ± 0.1 ^a
Ketones							
Propanone	81.9 ± 12.1 ^{ab}	186.1 ± 29.6 ^c	70.7 ± 7.0 ^a	79.1 ± 5.7 ^{ab}	104.4 ± 9.4 ^{ab}	79.3 ± 6.7 ^{ab}	118.6 ± 14.0 ^b
2,3-Butanedione	61.1 ± 10.4 ^a	75.1 ± 8.8 ^a	61.1 ± 6.0 ^a	91.2 ± 9.7 ^{ab}	120.8 ± 17.8 ^b	112.0 ± 20.4 ^{ab}	67.9 ± 9.18 ^a
Butanone	154.9 ± 18.6 ^a	183.7 ± 16.7 ^a	422.9 ± 41.6 ^b	441.8 ± 34.8 ^b	154.9 ± 13.4 ^a	170.7 ± 14.2 ^a	209.1 ± 20.2 ^a
2-Pentanone	161.0 ± 23.5 ^{ab}	128.3 ± 14.9 ^{ab}	405.2 ± 130.3 ^b	735.1 ± 97.6 ^c	80.6 ± 10.2 ^a	102.3 ± 11.0 ^a	233.4 ± 32.6 ^{ab}
3-Hydroxy-2-butanone	0	0	0	0	1.6 ± 1.6	19.5 ± 16.7	2.0 ± 1.0
3-Penten-2-one	0	0	9.3 ± 5.7	6.7 ± 0.8	1.0 ± 0.7	4.2 ± 2.3	8.8 ± 2.4
3-Methyl-2-pentanone	0	0	21.8 ± 11.4	0	0	0	0
3-Hexanone	0	0	16.9 ± 9.9	5.7 ± 5.7	0	0	2.9 ± 2.9
2-Hexanone	0.8 ± 0.3 ^a	2.3 ± 0.4 ^{ab}	39.4 ± 9.7 ^b	127.6 ± 16.3 ^c	1.7 ± 0.8 ^a	0.4 ± 0.2 ^a	23.6 ± 4.4 ^{ab}
Cyclohexanone	0 ^a	0 ^a	9.1 ± 1.8 ^b	0.9 ± 0.3 ^a	0.3 ± 0.3 ^a	0 ^a	0.6 ± 0.3 ^a
4-Heptanone	0 ^a	0 ^a	8.2 ± 3.6 ^b	1.0 ± 1.0 ^a	0 ^a	0 ^a	0 ^a

(Continued)

Table 2. —cont'd

Compound	Bayonne 1	Bayonne 2	Corsican	Iberian	LIC	Parma	Serrano
2-Heptanone	4.9 ± 2.6 ^a	12.4 ± 4.0 ^a	155.8 ± 31.2 ^b	376.0 ± 41.0 ^c	14.38 ± 4.2 ^a	6.9 ± 2.3 ^a	137.7 ± 18.6 ^b
4-Octen-3-one	0.1 ± 0.1	0.3 ± 0.1	1.2 ± 0.4	1.9 ± 0.9	0.3 ± 0.1	0.3 ± 0.1	0.5 ± 0.2
6-Methyl-5-hepten-2-one	6.0 ± 6.0	28.8 ± 16.1	43.1 ± 8.6	13.1 ± 6.4	24.7 ± 7.2	31.3 ± 8.0	21.5 ± 7.1
2-Octanone	6.8 ± 2.2 ^{ab}	0.1 ± 0.1 ^a	27.5 ± 4.5 ^b	100.3 ± 8.5 ^d	1.4 ± 0.4 ^a	2.71 ± 0.7 ^a	48.0 ± 5.7 ^c
8-Nonen-2-one	0	0	0	4.5 ± 1.3	0	0	3.8 ± 1.8
2-Nonanone	2.6 ± 0.8	0.8 ± 0.3	13.0 ± 7.9	11.8 ± 2.3	1.0 ± 0.2	1.4 ± 0.3	14.8 ± 6.9
Esters							
Ethyl acetate	1.0 ± 1.0 ^a	97.9 ± 13.1 ^b	0 ^a	1.8 ± 1.0 ^a	0 ^a	0 ^a	58.9 ± 21.9 ^b
Ethyl propanoate	0 ^{ab}	0 ^a	0 ^a	0.1 ± 0.1 ^a	0.2 ± 0.2 ^a	0 ^a	5.3 ± 2.8 ^b
Ethyl 2-methyl propanoate	0.3 ± 0.2 ^a	21.0 ± 2.8 ^b	2.6 ± 0.7 ^{ab}	0.1 ± 0.1 ^a	0.7 ± 0.1 ^a	0.3 ± 0.1 ^a	12.8 ± 6.3 ^b
Ethyl 3-methyl butanoate	0.7 ± 0.5 ^a	20.2 ± 2.1 ^b	4.7 ± 1.4 ^{ab}	1.1 ± 0.2 ^a	1.3 ± 0.3 ^a	0.5 ± 0.2 ^{ab}	18.1 ± 8.5
1-Penten-3-ol acetate	0.2 ± 0.1 ^a	45.7 ± 4.9 ^b	12.4 ± 2.6 ^{ab}	2.5 ± 0.5 ^a	3.5 ± 0.6 ^a	2.5 ± 0.6 ^a	39.8 ± 19.3 ^b
Methyl hexanoate	0	1.01 ± 0.5 ^{ab}	2.5 ± 0.7 ^b	0.1 ± 0.1 ^a	0.7 ± 0.2 ^a	1.2 ± 0.3 ^{ab}	0.1 ± 0.1 ^a
Ethyl hexanoate	0.4 ± 0.4 ^a	0.3 ± 0.3	0.4 ± 0.2	0.6 ± 0.3	0	0.1 ± 0.1	0.8 ± 0.3
Pentyl butanoate	0	30.1 ± 4.4 ^{ab}	10.6 ± 2.2 ^a	18.6 ± 2.7 ^a	4.2 ± 0.9 ^a	2.4 ± 0.7 ^a	59.8 ± 19.9 ^b
Ethyl heptanoate	0 ^a	0	0.4 ± 0.2	0	0	0	0
Ethyl octanoate	2.6 ± 1.2	0.2 ± 0.2 ^a	0 ^a	0.6 ± 0.2 ^a	0 ^a	0 ^a	3.2 ± 1.4 ^b
		4.4 ± 1.7	3.1 ± 0.7	3.4 ± 0.5	2.1 ± 0.4	3.08 ± 0.8	3.4 ± 0.8
Nitrogenous compounds							
N-Methylene ethenamine	0 ^a	0 ^a	0.9 ± 0.5 ^{ab}	0.7 ± 0.4 ^{ab}	0 ^a	0 ^a	1.4 ± 0.4 ^b
Pyrrrol	11.9 ± 4.3 ^b	1.1 ± 1.1 ^a	16.0 ± 4.0 ^b	0.6 ± 0.5 ^a	0.2 ± 0.2 ^a	0.1 ± 0.1 ^a	1.1 ± 0.8 ^a
Methyl pyrazine	0	0	2.9 ± 2.9	0	0	0	0
Hexanenitrile	0 ^a	0 ^a	9.0 ± 2.9 ^{ab}	18.8 ± 5.0 ^b	0 ^a	0 ^a	10.4 ± 4.2 ^{ab}
2,6-Dimethyl pyrazine	0	0	298.4 ± 144.4	0	0	0	0
Trimethyl pyrazine	0	0	36.3 ± 18.3	0	0	0	0
3-Ethyl-2,5-dimethyl pyrazine	0	0	12.7 ± 7.8	0	0	0	0
2-Ethyl-2,5-dimethyl pyrazine	0	0	7.9 ± 4.6	0	0	0	0
2,3-Diethyl-5-methyl pyrazine	0	0	0.5 ± 0.4	0	0	0	0
3,5-Diethyl-2-methyl pyrazine	0	0	1.5 ± 1.0	0	0	0	0
n-Alkanes							
Hexane	97.2 ± 37.2	44.5 ± 29.6	140.2 ± 42.5	252.7 ± 90.2	67.4 ± 19.2	65.5 ± 23.5	83.8 ± 28.1
Heptane	54.7 ± 19.4 ^{ab}	49.3 ± 24.5 ^{ab}	9.8 ± 5.48 ^a	3.8 ± 2.7 ^a	41.6 ± 12.2 ^{ab}	66.8 ± 13.1 ^b	36.0 ± 13.6 ^{ab}
Octane	170.2 ± 45.1	214.7 ± 54.7	232.1 ± 58.1	505.7 ± 283.5	126.7 ± 37.9	94.9 ± 12.8	143.0 ± 20.9
Nonane	2.0 ± 0.5 ^a	1.4 ± 0.5 ^a	6.1 ± 0.8 ^b	3.6 ± 1.3 ^{ab}	1.5 ± 0.4 ^a	1.6 ± 0.4 ^a	2.7 ± 0.7 ^a
Undecane	2.23 ± 0.6	3.0 ± 0.7	3.6 ± 0.6	2.7 ± 0.4	2.0 ± 0.4	2.6 ± 0.5	3.0 ± 1.2
Dodecane	5.1 ± 1.1	6.0 ± 0.8	5.9 ± 0.6	5.6 ± 0.6	4.6 ± 0.8	4.0 ± 0.6	8.7 ± 3.9
Tridecane	3.5 ± 1.5 ^a	1.1 ± 0.2 ^a	2.6 ± 1.4 ^a	20.6 ± 4.6 ^b	3.0 ± 1.0 ^a	2.7 ± 1.4 ^a	4.7 ± 0.8 ^a
Aromatic and cyclic hydrocarbons							
Methyl cyclopentane	56.2 ± 10.7 ^{ab}	103.7 ± 24.7 ^{ab}	70.3 ± 15.4 ^{ab}	112.6 ± 39.1 ^b	29.2 ± 6.5 ^a	30.5 ± 8.1 ^a	60.5 ± 12.9 ^{ab}
Toluene	8.6 ± 2.3 ^{ab}	6.1 ± 1.5 ^a	21.5 ± 5.2 ^c	17.7 ± 1.1 ^{bc}	5.2 ± 0.7 ^a	7.5 ± 1.2 ^a	11.4 ± 1.4 ^{ab}
Ethyl benzene	5.2 ± 1.2 ^a	4.4 ± 1.8 ^a	10.4 ± 1.3 ^b	5.1 ± 0.3 ^a	4.9 ± 0.5 ^a	5.4 ± 0.5 ^a	7.3 ± 1.3 ^{ab}
Dimethyl benzene	8.4 ± 2.5 ^{ab}	5.8 ± 1.4 ^a	13.2 ± 1.7 ^b	8.2 ± 0.8 ^{ab}	7.0 ± 0.9 ^a	8.2 ± 1.3 ^{ab}	8.3 ± 1.1 ^{ab}
Dimethyl benzene	5.0 ± 1.3 ^a	2.0 ± 0.1 ^a	43.1 ± 20.0 ^a	126.3 ± 35.2 ^b	4.2 ± 0.7 ^a	5.0 ± 0.8 ^a	43.5 ± 17.9 ^a

(Continued)

Table 2. —*cont'd*

Ethyl toluene	0.8±0.3 ^a	36.8±26.9 ^{ab}	18.7±6.3 ^a	56.8±12.1 ^b	5.4±4.7 ^a	6.0±4.0 ^a	19.2±5.5 ^a
Trimethyl benzene	0 ^a	0.6±0.6 ^a	1.5±0.9 ^a	6.0±1.4 ^b	0.1±0.1 ^a	0 ^a	0.3±0.3 ^a
1-Ethyl-2-methyl cyclopentane	0	0	12.8±2.8 ^b	6.6±1.4 ^{ab}	0.9±0.4 ^a	1.3±1.1 ^a	6.2±1.2 ^a
Trimethyl benzene	2.3±1.6	0	0.8±0.8	0	0	1.9±1.1	0
Sulphur compounds							
Diethyl benzene	0 ^a	0.5±0.2 ^a	1.5±0.3 ^b	0.2±0.1 ^a	0.2±0.1 ^a	0.1±0.1 ^a	0 ^a
1,2,2-Trimethyl benzene	0.2±0.1 ^a	4.7±1.8 ^b	1.5±0.3 ^a	0.8±0.5 ^a	0.2±0.1 ^a	0.4±0.2 ^a	0.3±0.3 ^a
etramethyl benzene	0.4±0.2 ^a	6.4±2.0 ^c	3.2±0.7 ^b	0.3±0.1 ^a	0.4±0.1 ^a	0.5±0.2 ^a	0 ^a
Sulphur compounds							
Carbon disulphide	4.5±1.1	10.9±2.3	4.5±0.9	5.9±1.8	4.8±0.7	4.7±1.1	4.7±1.1
Dimethyl disulphide	97.0±11.6 ^a	121.1±27.5 ^a	297.0±54.28 ^b	159.6±18.2 ^a	81.8±13.1 ^a	70.2±8.8 ^a	172.7±19.4 ^a
Dimethyl trisulphide	0.4±0.3 ^a	0.7±0.7 ^a	31.1±5.0 ^b	23.9±3.9 ^b	0 ^a	0.8±0.5 ^a	10.8±2.7 ^a
Methyl <i>n</i> -pentyl disulphide	0.1±0.1	0.4±0.2	0.7±0.4	0.8±0.2	0.1±0.1	0.1±0.1	0.6±0.1
Methyl <i>n</i> -hexane disulphide	0.3±0.1	2.7±1.6	3.0±0.5	1.5±0.3	0.2±0.2	0.3±0.1	7.1±4.0
Terpenes							
Unknown terpene	2.6±1.4 ^a	40.4±12.4 ^c	22.8±3.4 ^b	3.7±0.9 ^a	1.7±0.4 ^a	2.7±0.7 ^a	2.3±0.8 ^a
Canphene	0.3±0.2 ^a	3.29±1.0 ^b	0.7±0.3 ^a	0.1±0.1 ^a	0.1±0.1 ^a	0.3±0.2 ^a	0 ^a
β -Phelandrene	0	6.1±2.6	5.5±3.3	0.1±0.1	0	0	0.1±0.1
β -Pinene	0.2±0.2 ^a	54.2±17.7 ^b	11.1±3.6 ^a	0 ^a	0 ^a	0.3±0.2 ^a	0.2±0.1 ^a
3-Carene	0.6±0.3 ^a	54.4±17.7 ^b	11.3±3.6 ^a	0.6±0.2 ^a	0.1±0.1 ^a	0.8±0.3 ^a	0.4±0.2 ^a
2,3-Dihydrocineol	0	0	38.4±20.9	0	0	0	0
β -Myrcene	0	22.3±9.4	0	0	0	0	0
α -Pinene	0	3.5±2.4	1.8±1.8	0	0	0	0.6±0.6
α -Phelandrene	0 ^a	105.9±37.6 ^b	30.7±11.5 ^a	0 ^a	0 ^a	0 ^a	0 ^a
2-Carene	0 ^a	173.5±58.6 ^b	46.6±14.5 ^a	0 ^a	0 ^a	0 ^a	0 ^a
Unknown terpene	0 ^a	1.2±0.4 ^b	0 ^a	0 ^a	0 ^a	0 ^a	0 ^a
Limonene	3.5±1.3 ^a	97.2±32.2 ^b	20.2±6.1 ^a	9.7±2.4 ^a	2.2±0.5 ^a	4.7±1.2 ^a	7.9±3.8 ^a
Cineol	1.4±1.0	0	0	0	0.5±0.4	1.6±1.0	0
Unknown terpene	0.1±0.1	2.3±0.7	2.6±1.6	4.8±4.8	0.4±0.2	0.7±0.3	0.1±0.1
Unknown terpene	2.3±0.6	2.2±0.9	6.7±2.6	2.2±0.4	2.2±0.5	3.2±0.6	6.6±1.1
Unknown terpene	0.2±0.2 ^a	2.2±0.6 ^b	0.1±0.1 ^a	0 ^a	0.1±0.1 ^a	0.8±0.5 ^a	0 ^a
Unknown terpene	0.5±0.3 ^a	5.0±1.6 ^b	0.8±0.3 ^a	0 ^a	0.1±0.1 ^a	0.8±0.5 ^a	0 ^a
Caryophyllene	0.3±0.2 ^a	19.9±5.4 ^b	1.6±0.9 ^a	0 ^a	0.9±0.2 ^a	2.0±0.4 ^a	0 ^a

Means in the same row with a different letter are significantly different at the 0.05 level. LIC, Light Italian Country.

high level of terpenes. As far as we know, this is the first time that the high capacity for diffusion of some volatile compounds in ham (the biceps femoris is an inner muscle) has been demonstrated. The main consequence of this diffusion is that any effect at the surface of the ham (e.g. adding of additive, covering with fat, growing of mould, etc.) may have important sensory implications.

Nitrogenous compounds

Some Corsican hams have a very high level of pyrazines, mainly 2,6-dimethyl pyrazine. Pyrazines are compounds found in many meats and meat products prepared by cooking at high temperature (Mussinan and Walradt, 1974). The high temperature promotes the reaction between diketo compounds and amino compounds, leading to pyrazine formation (Shibamoto and Bernhard, 1976). However, during dry-cured ham processing low temperatures are used (< 30°C). The dehydration process may favour this reaction, but it may lead to the presence of only a small amount of pyrazine. Probably, the high level of these compounds found in some Corsican hams comes from an external agent (an additive such as paprika, smoke, etc.) (Tóth and Potthast, 1984).

Principal components

Principal component analysis allows one to obtain a better overall idea of the behaviour of data and variables. The percentages of total variance explained by the first four components are 23.3, 14.2, 10 and 7.5%, respectively.

Figure 1 shows the variable loading and average score of each type of hams on the plane of the two first components. Only the variables with loading higher than 0.4 are presented. Component 1 was determined in decreasing order of importance by: (1) medium-chain linear aldehydes (pentanal, hexanal, and heptanal), medium-chain methyl ketones (2-hexanone, 2-heptanone, and 2-octanone), and hexanol; (2) branched aldehydes (2-methylbutanal, 3-methylbutanal, and 2-methylpropanal), octanal, 2,4-nonadienal, short-chain methyl ketones (2-butanone and 2-pentanone), and secondary alcohols (2-pentanol and 2-heptanol).

In all these compounds, the contribution to component 2 is very low. The distribution in the plane PC1–PC2 shows that these volatile compounds are quite clustered, which indicates that they are highly correlated. In fact, the formation pathway in most of these compounds is related to oxidation.

On the other hand, component 2 was determined by:

1. ethyl esters (ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, ethyl hexanoate, ethyl acetate, and ethyl 2-methylpropanoate) and 2-hexanol;
2. short-chain compounds (2-propanol, 2-butanol, 2-propanone, and acetaldehyde).

These compounds are correlated, and they are independent of component 1. Probably, in the formation pathways of most of these compounds micro-organisms are involved.

The distribution of ham type in the PC1–PC2 plane agrees with the ANOVA results. Iberian ham is much richer in volatile compounds, which come from the

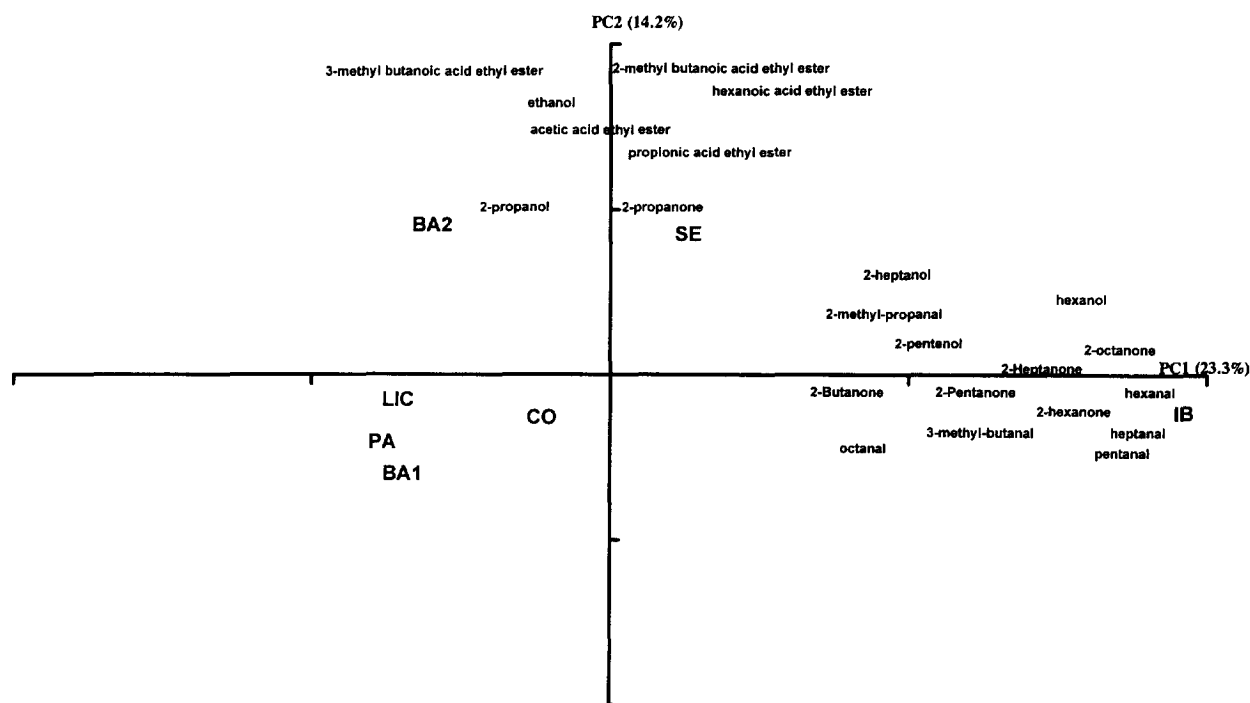


Fig. 1. Variable loading and average score of each type of ham plot from PC analysis on content of volatile compounds. PC1–PC2 plane.

oxidation of lipids and amino acids, than Bayonne, Parma, Light Italian Country, and Corsican hams. Serano hams are intermediate. This difference must be due to both the higher intramuscular fat content in Iberian hams and the high temperatures used in drying. The high temperature probably increases: (1) the proteolytic and lipolytic processes, raising the amount of free fatty acids and free amino acids, which are the precursors of these compounds; and (2) the rates of reactions involved in the oxidation process.

Figure 2 shows the variable loading and average score of each type of ham on the plane defined by components 3 and 4. Component 3 was determined, in decreasing order of importance, by:

1. 1-penten-3-ol, benzaldehyde, octanol, and dimethylsulphide;
2. 2-butanone, 2,6-dimethylpyrazine, 2-methylpropanal, nonanal and decanal, dimethyltrisulphide.

The results indicate that these compounds are characteristic of Corsican hams. Component 4 is clearly defined by terpenes. As the ANOVA indicates, terpenes are abundant in group 2 of Bayonne hams. Nevertheless, sensory analysis did not show any significant difference between Bayonne 1 and Bayonne 2, which indicates that the effect of the terpenes is low at these concentrations.

Discriminant analysis

In order to verify the capacity of volatile compound analysis as a tool for ham type discrimination, a Stepwise Discriminant Analysis was carried out. The program selects 44 variables and calculates six discriminant functions, which classify, correctly, 84% of the total cases. The classification results are shown in Table 3. Most of the groups, especially Bayonne 2, Corsican and Iberian hams, were very well classified, but it was very difficult to distinguish between Parma and Light Italian Country hams.

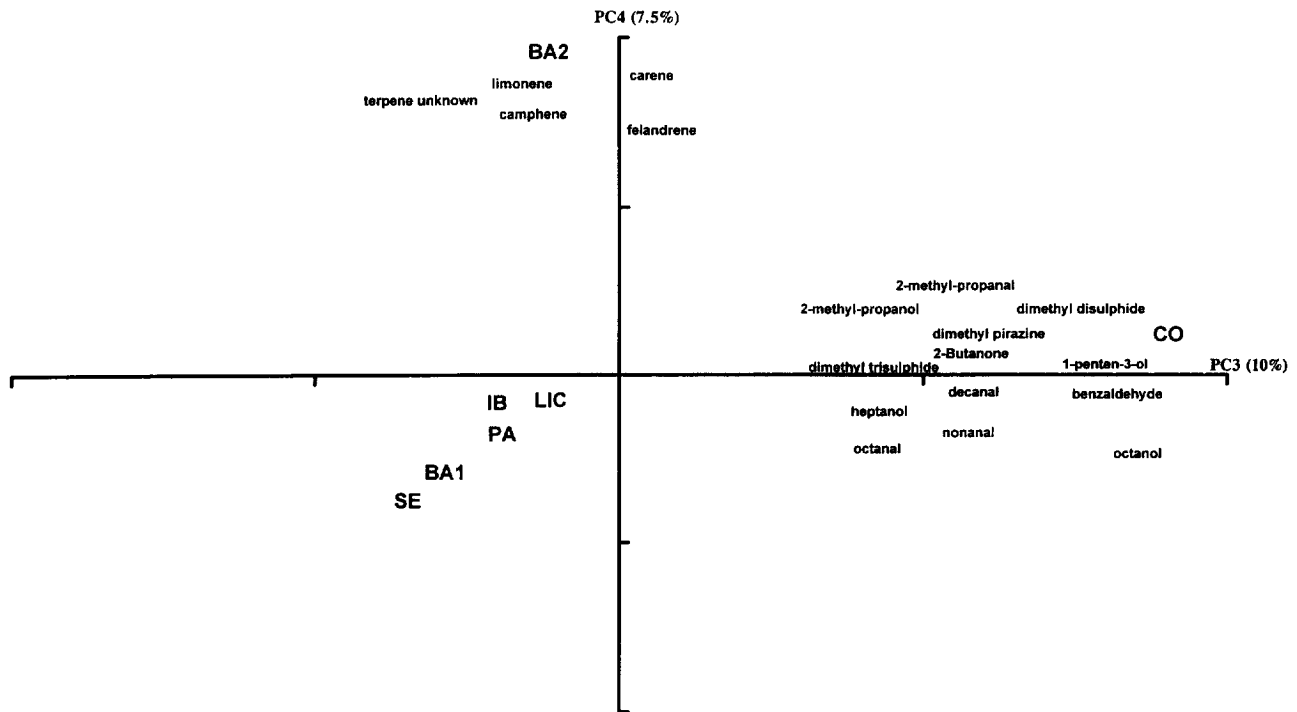


Fig. 2. Variable loading and average score of each type of ham plot from PC analysis on content of volatile compounds. PC3-PC4 plane.

Table 3. Percentage classification results of discriminant analysis

Actual group	Predicted group (%)						
	BA1	BA2	CO	IB	LIC	PA	SE
BA1	86.7	0	0	0	0	13.3	0
BA2	0	100	0	0	0	0	0
CO	0	0	96.7	3.3	0	0	0
IB	3.3	0	0	93.3	0	3.3	0
ICS	3.3	0	0	0	66.7	30.0	0
PA	20.0	0	0	0	10.0	70.0	0
SE	10.0	0	0	0	0	6.7	83.3

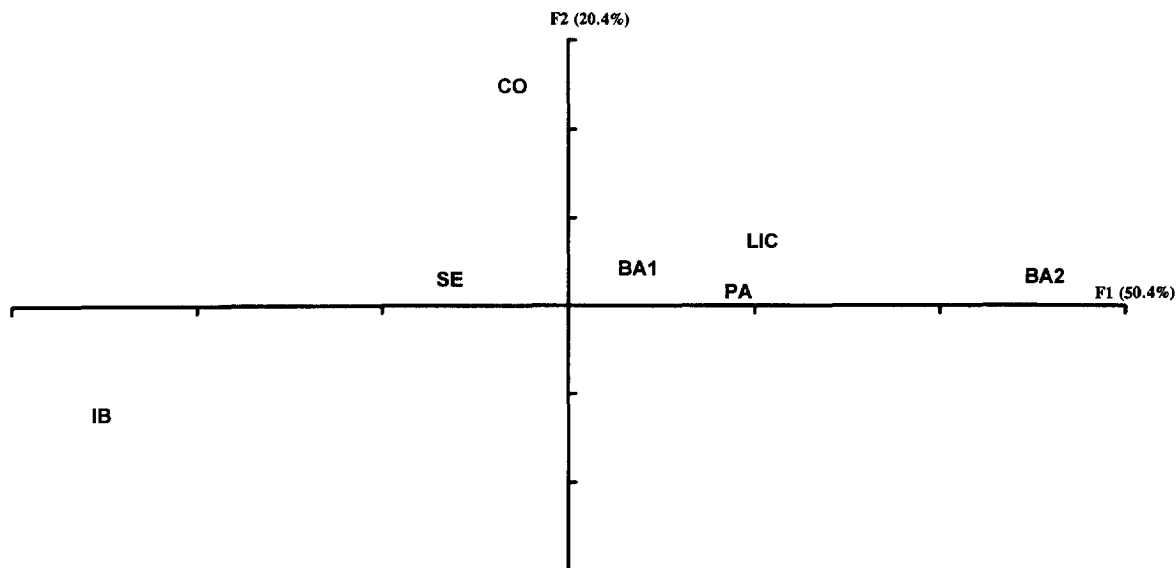


Fig. 3. Stepwise discriminant analysis results.

Figure 3 shows the plane F1–F2, which accumulates 70.8% of the total variance. This figure emphasises the importance of the different technologies on the volatile compound profiles of hams. The results agree with ANOVA and PCA, and they indicate that Parma, Light Italian Country hams and Bayonne 1 (the three with similar processing) are quite similar products, while Iberian and Corsican are very different. Serrano seems to be intermediate between the rawer hams (Parma, Light Italian Country hams and Bayonne) and Iberian. In fact, Serrano ham technology has some similarity to Iberian; however, it is medium-time processing while Iberian is long-time processing. Moreover, Iberian ham has a higher amount of intramuscular fat, which is a precursor of a large number of volatile compounds. Finally, Bayonne 2 appears different from Parma, Light Italian Country and Bayonne 1 ham, owing to the large number of terpenes.

CONCLUSIONS

There are important differences between the six types of hams, depending on the type of raw matter and the technology used, the long-time processed hams (Iberian and Corsican) being richer in volatile compounds than the others. The six types of hams can be classified into three groups:

Iberian ham, which is characterized by (1) a high amount of linear medium-chain compounds, mainly aldehydes and methyl ketones, and, to a lesser extent, alcohols. All these compounds come from lipid degradation; (2) branched aldehydes, that come from amino acid degradation.

Corsican ham, which is characterized by: (1) branched aldehydes, (2) unsaturated alcohols, (3) no-methyl

ketones, (4) terpenes, (5) sulphur compounds, and (6) nitrogenous compounds, mainly pyrazines.

Bayonne, Light Italian Country and Parma. These rawer hams are characterized, in general, by a smaller amount of volatile compounds than Iberian and Corsican ones. Serrano ham seems to be intermediate between Iberian and the group of rawer hams. Bayonne 2 ham is rich in terpenes owing to the presence of pepper at the surface.

Most of the volatile compounds found in dry-cured hams come from lipid autoxidation and amino acid degradation, aldehydes being the most abundant family. The high levels of terpenes found in some hams is due to the application of pepper at the ham surface.

Finally, the results of the Stepwise Discriminant Analysis show that the analysis of volatile compounds may be a useful tool for ham certification.

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