

## Volatile compounds in Iberian dry-cured loin

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### Abstract

The volatile profile of Iberian dry-cured loin from four different Iberian pig lines (Entrepelado, Lampiño, Retinto and Torbiscal) and two feeding systems (OUT – fed on acorn and grass-vs.-IND – fed on high oleic acid concentrate) was studied using solid phase microextraction (SPME). 133 volatile compounds were identified and assigned to 16 chemical families. Alcohols were the major group, ethanol being the main compound. The high number of esters detected and the levels of ethanol and acetic acid found, points to an important role of microorganism activity in the formation of volatile compounds in Iberian dry-cured loin. Sulphur compounds, coming mostly from garlic, constituted an important group, with 14 compounds. Significant differences were found among loins from pigs reared in different feeding systems but not among Iberian pig lines. Dry-cured loins from OUT pigs showed higher levels of many compounds derived from lipid oxidation, such as octanoic acid ( $P = 0.000$ ), decanoic acid ( $P = 0.018$ ) or hexanal ( $P = 0.014$ ).

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### 1. Introduction

The overall acceptance of meat products depends to a large extent on their flavour, which is mainly determined by taste and odour compounds (Ramarathnam & Rubin, 1994). In dry-cured products, chemical and biochemical changes during ripening give rise to a large number of volatile compounds which contribute to their characteristic flavour (Ruiz, Muriel, & Ventanas, 2002). Moreover, compounds from feeds accumulated in fat depots of the animal and compounds coming from ingredients also contribute to the final flavour of dry-cured meat products.

Iberian dry-cured loin is one of the most valuable meat products in Spain. It is produced by rubbing a mixture of curing agents (salt and nitrite) and spices (Spanish paprika, *Capsicum annuum*, L. and garlic, *Allium sativum*, L.) onto the surface of the loin pieces. The

loins are then stuffed into casings and subsequently undergo the drying/ripening process.

Quality of meat products from Iberian pigs strongly depends upon breed and rearing system. The Iberian pig breed includes a number of different lines, causing a great heterogeneity within the same breed. Several authors have reported important differences in productive, reproductive and morphologic parameters between different lines (Benito et al., 1998). However, the influence of Iberian pig line on the physical, chemical and sensory characteristics of dry-cured loin remains unstudied.

Concerning the rearing system, products from Iberian pigs raised in the traditional fattening system, in which animals are free reared outdoors with free availability of acorn and grass, attain the highest quality and prices (Carrapiso, Jurado, Timón, & García, 2002). However, most Iberian pigs are nowadays reared in confinement and fed on concentrates. In the last decade, the formulation of concentrates for Iberian pig feeding has changed in order to imitate the fat composition of meat from pigs reared on acorns and grass. Thus, the use of concentrates rich in oleic acid for Iberian pig feeding is

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increasing (Muriel, Ruiz, Ventanas, & Antequera, 2002). However, the effect of this type of feeding on the characteristics of dry-cured products from Iberian pigs has not been studied yet.

Many works have been published about the volatile profiles of different dry-cured hams and different kinds of dry-cured sausages (García et al., 1991; Mateo & Zumalacárregui, 1996; Schmidt & Berger, 1998). However, there are only few studies regarding dry-cured loins (Antequera, Muriel, Rodríguez, Cernadas, & Ruiz, 2004; Muriel, Ruiz, Ventanas, Petró, & Antequera, 2004) and no papers describing its volatile profile.

This study deals with the description of the volatile profile of Iberian dry-cured loin and the influence of Iberian pig line and the feeding system during the fattening period on such profile.

## 2. Materials and methods

### 2.1. Experimental design

This study was carried out with 40 pure Iberian pigs from four different lines (Entrepelado, Lampiño, Retinto and Torbiscal, 10 animals each line). During the fattening period (60 days previous to slaughter) half the animals of each line were raised outdoors in a 30 Ha extension land, the exclusive feed source being acorn and grass (OUT). The other 20 were raised in a 6 Ha extension land and fed on a high oleic acid (C18:1,  $n-9$ ) concentrate (IND). Chemical composition of feeds has been published in a previous paper (Muriel et al., 2002). All animals were slaughtered at  $150 \pm 10$  kg by electrical stunning and exsanguination at a local slaughterhouse. Sampling was carried out within the hour following slaughter. The cranial half of the right loin of all 40 animals was taken and processed at a commercial plant. Loins were seasoned with a pickling sauce (Table 1) containing salt, nitrites, Spanish paprika, garlic and olive oil. Then, loins were kept for 3 days at 3 °C, to allow the seasoning mixture to penetrate. After this time, loins were stuffed into artificial casings and held for 90 days at 6 °C with a relative humidity around 85%. Once the processing was completed, loins were vacuum packaged and kept frozen ( $-80$  °C) until analysis.

Table 1  
Ingredients and additives used in the pickling sauce

Ingredients and additives	Quantity, g (per kg of raw loin)
Dry salt	24
Nitrites	9
Bitter-sweet paprika	2
Garlic	1
Olive oil	5

### 2.2. Volatile analysis

Volatile compounds of dry-cured loins were extracted by headspace-SPME (Ruiz, Ventanas, & Cava, 2001). Dry-cured loin slices were ground in a domestic grinder. 1 g was weighed into a 4 ml vial and screw-capped with a Teflon-silicone disk. A SPME (Supelco, Bellefonte, PA, USA) fibre coated with carboxen/poly(dimethylsiloxane) (75 µm thickness) was inserted into the sample vial through the septum and then exposed to the headspace. Extraction was performed at 37 °C for 30 min in a water bath. The type of SPME fibre was chosen because of its high sensitivity and wide linear range of response (Brunton, Cronin, Monahan, & Duncan, 2000). Prior to analysis the SPME fibre was preconditioned at 280 °C for 50 min in the gas chromatography injection port.

Volatile compounds analysis was performed using a HP-6890-GC series II gas chromatograph (Hewlett-Packard) coupled to a mass selective detector (HP-5973 Network, Hewlett-Packard). Volatiles were separated using a 5% phenyl-methylsilicone (HP-5) bonded phase fused-silica capillary column (50 m × 0.32 mm i.d., film thickness 1.05 µm, Hewlett-Packard). Carrier gas was Helium at a flow rate of 1.45 ml min<sup>-1</sup>. The SPME fibre was desorbed and maintained in the injection port at 280 °C during the whole chromatographic run. A 0.75 mm i.d. glass liner was used. The injection port was in splitless mode. The temperature program was isothermal for 10 min at 40 °C, raised to 200 °C at a rate of 5 °C min<sup>-1</sup>, and held for 5 min. The transfer line to the mass spectrometer was maintained at 280 °C. The mass spectra were obtained by electronic impact at 70 eV, a multiplier voltage of 1756 V and collecting data at a rate of 1 scan s<sup>-1</sup> over the  $m/z$  range of 30–500. Compounds were tentatively identified by comparing their mass spectra with those contained in the NIST and Wiley libraries and by comparison of Kovats indexes with those reported in the literature (Kondjoyan & Berdagué, 1996). Results from volatile analyses are expressed as percentage of total chromatographic area.

### 2.3. Statistical analysis

The effect of Iberian pig line (Entrepelado, Lampiño, Retinto and Torbiscal) and feeding system (OUT vs. IND) on the proportion of identified volatiles was carried out by an analysis of variance (ANOVA), using de General Linear Model (GLM) procedure (SPSS v.11.0).

## 3. Results and discussion

### 3.1. Volatile profile of Iberian dry-cured loin

Fig. 1 shows a total ion chromatogram of the volatile compounds from Iberian dry-cured loin. A total of 126

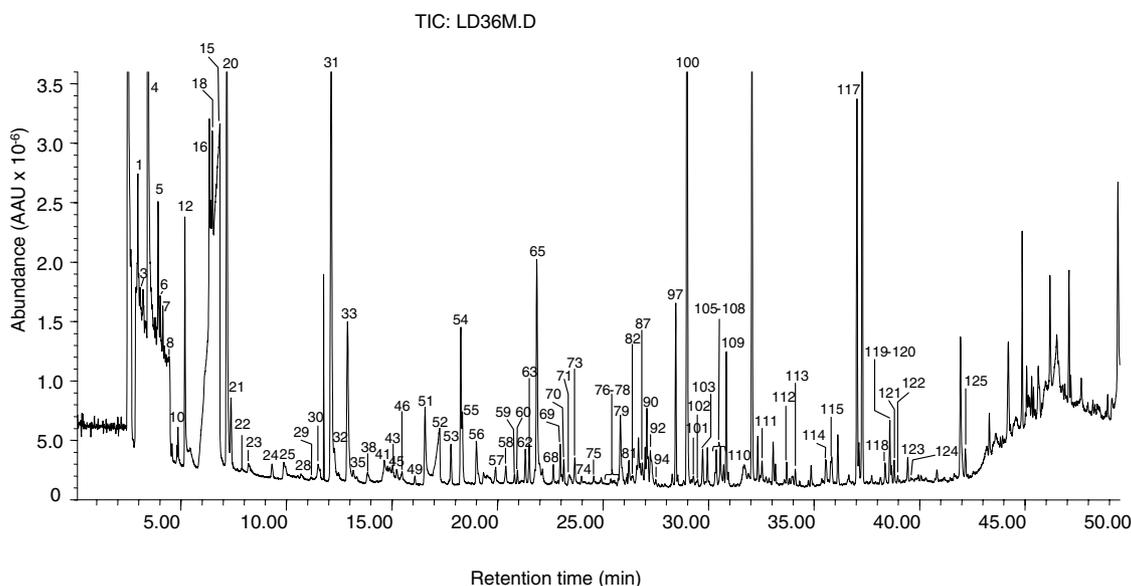


Fig. 1. Total ion current gas chromatogram of extracted volatile from one dry-cured Iberian loin sample. For peak numbers see Table 2.

peaks were identified, comprising 133 compounds. They were assigned to the following chemical families: acids (12), alcohols (22), aldehydes (11), aliphatic hydrocarbons (13), aromatic hydrocarbons (9), chloride compounds (1), esters (14), ethers (1), furans (6), ketones (9), nitrogen compounds (7), phenols (3), sulphur compounds (14) and terpenes (12). Thirteen compounds were detected in co-elution. These compounds have been classified into the family of the most abundant compound of those co-eluted, which has been situated at the first position.

As can be observed in the chromatogram shown in Fig. 1, there are a number of peaks that are not included in the list of volatile compounds (Table 2). Most of these peaks were tentatively identified as siloxanes or silanes, and its most probable origin is the SPME fibre, although some could also come from the chromatographic column. Some of the late eluting peaks could not be identified. However, their spectra suggest long chain aliphatic hydrocarbons as their most probable group.

Alcohols were quantitatively and qualitatively the most important chemical family in Iberian dry-cured loin, accounting for at least the 35% of total chromatographic area, followed by acids and ketones, with 18% and 12% of total chromatographic area, respectively. Individually, the major compounds were ethanol, acetic acid, 2-butanone, 2-butanol, hexane and (E)-3-(methylthio)-propene.

A high number of acids have been detected. This agrees with previous research on Iberian dry-cured ham using SPME (Andrés, Cava, & Ruiz, 2002). Acetic was the most abundant acid. Its origin is mainly the microbial metabolism of glucides (Kandler, 1983). The rest of the straight chain aliphatic acids could derive from the

hydrolysis of triglycerides and phospholipids, although most of them arise from lipid oxidation reactions (Ruiz et al., 2002). On the other hand, branched chain acids such as 2-methylpropanoic acid, 3-methylbutanoic acid and 2-methylbutanoic acid, have been identified as products of microbial metabolism of valine, leucine and isoleucine, respectively (Stahnke, 1995a), thus these compounds could arise from the microorganisms activity on the surface of the loins. Nevertheless, Mateo, Aguirrezábal, Domínguez, and Zumalacárregui (1997) found some of these acids (propanoic acid, 2-methylpropanoic acid, butanoic acid, 3-methylbutanoic acid and 2-methylbutanoic acid) among the volatile compounds of Spanish paprika, which was one of the spices used in the pickling sauce of the loins. Octanoic, nonanoic, decanoic and dodecanoic acids, can act as precursors of compounds affecting taste or aroma, but most likely, do not directly influence the flavour of cured products due to their high olfaction threshold (Belitz & Grosch, 1997). However, short chain acids (<6 carbon atoms) have an important effect in aroma development due to their characteristic odours, described as vinegar, cheese or cucumber (Stahnke, 1995b) and to their low threshold values (Belitz & Grosch, 1997). In this sense Schmidt and Berger (1998) found 3-methylbutanoic acid to be a key compound among the odour-active compounds in different types of dry-cured sausages.

Regarding the alcohols, the high levels of ethanol found have been previously described in French dry-cured sausage (Croizet, Denoyer, Tran, & Berdagué, 1992), though these authors found this compound did not contribute significantly to the product aroma. Straight chain alcohols come from polyunsaturated fatty acid oxidation (Forss, 1972), although low

Table 2  
Percentage of area relative to the total peak area of the identified peaks of the volatile compounds in dry-cured Iberian loin from pigs on different feeding systems

Peak No.	Compounds	Feeding system					
		KI	Id	OUT	IND	P	SEM
	<i>Acids</i>			18.95	17.33	0.235	0.674
15	Acetic acid	592	MS/KI	16.32	14.60	0.223	0.700
46	2-Methylpropanoic acid	751	MS/KI	0.09	0.17	0.020	0.018
52	Butanoic acid + 2,3-butanediol	776	MS/KI	1.09	0.92	0.210	0.070
57	3-Methylbutanoic acid	831	MS/KI	0.25	0.28	0.754	0.048
58	2-Methylbutanoic acid	840	MS/KI	0.17	0.21	0.475	0.024
75	4-Methylpentanoic acid	938	MS	0.08	0.10	0.180	0.009
79	Hexanoic acid + benzaldehyde	969	MS/KI	0.67	0.88	0.390	0.120
111	Octanoic acid	1147	MS/KI	0.06	0.04	0.000	0.004
114	Nonanoic acid	1249	MS/KI	0.05	0.02	0.000	0.004
118	Decanoic acid	1357	MS/KI	0.10	0.06	0.001	0.007
126	Dodecanoic acid	1556	MS/KI	0.06	0.06	0.882	0.007
	<i>Alcohols</i>			31.71	36.15	0.186	1.662
4	Ethanol	<500	MS	16.80	18.09	0.600	1.203
11	2-Propen-1-ol (Allyl alcohol)	536	MS	0.04	0.05	0.799	0.004
12	1-Propanol + 2-methylpropanal	547	MS/KI	2.85	3.08	0.829	0.535
19	2-Butanol	602	MS/KI	7.63	9.38	0.274	0.789
22	2-Methylpropanol	621	MS/KI	0.34	0.46	0.218	0.050
23	2-Methoxyethanol	630	MS	0.36	0.31	0.687	0.054
25	1-Butanol + 2-methylbutanal + benzene	658	MS/KI	0.59	1.20	0.001	0.098
28	1-Methoxy-2-propanol	672	MS/KI	0.07	0.11	0.024	0.009
29	1-Penten-3-ol	680	MS/KI	0.06	0.06	0.989	0.007
34	2-Ethoxyethanol	714	MS	0.16	0.19	0.552	0.023
40	3-Methyl-1-butanol	735	MS/KI	1.56	1.69	0.813	0.277
42	2-Methyl-1-butanol	739	MS/KI	0.42	0.55	0.495	0.093
50	1-Pentanol	766	MS/KI	0.16	0.16	0.812	0.015
64	1-Hexanol	868	MS/KI	0.20	0.23	0.624	0.028
67	Cyclohexanol	887	MS/KI	0.12	0.21	0.003	0.016
71	2-Butoxyethanol	908	MS/KI	0.10	0.16	0.004	0.010
82	1-Octen-3-ol	980	MS/KI	0.08	0.04	0.000	0.007
96	2-Ethyl-1-hexanol	1028	MS/KI	0.02	0.02	0.947	0.002
110	Benzeneethanol	1114	MS/KI	0.16	0.15	0.981	0.018
	<i>Aldehydes</i>			8.13	5.08	0.001	0.482
1	Acetaldehyde	<500	MS	0.22	0.24	0.698	0.031
24	3-Methylbutanal	649	MS/KI	0.36	0.49	0.205	0.049
54	Hexanal	795	MS/KI	2.00	0.57	0.007	0.276
70	Heptanal	902	MS/KI	0.19	0.10	0.036	0.022
92	Octanal	1004	MS/KI	0.08	0.04	0.001	0.006
100	Benzeneacetaldehyde	1050	MS/KI	4.40	3.33	0.098	0.324
109	Nonanal	1099	MS/KI	0.77	0.28	0.000	0.075
121	2-Methylundecanal	1372	MS	0.10	0.04	0.001	0.010
	<i>Alifatic hydrocarbons</i>			8.40	3.87	0.056	1.188
2	2-Methylpropene	<500	MS	0.08	0.15	0.029	0.016
6	Pentane	500	MS/KI	0.20	0.52	0.040	0.080
13	2-Methylpentane	553	MS/KI	0.19	0.29	0.080	0.028
14	3-Methylpentane	577	MS/KI	0.12	0.15	0.361	0.015
18	Hexane	600	MS/KI	6.22	2.05	0.089	1.222
28	3-Methylhexane	672	MS/KI	0.06	0.07	0.371	0.006
32	Heptane	700	MS/KI	0.17	0.19	0.756	0.039
88	Branched alkane	996	MS	0.05	0.06	0.315	0.006
90	Decane	1000	MS/KI	1.06	0.07	0.030	0.230
98	Butylcyclohexane	1038	MS/KI	0.10	0.12	0.646	0.012
116	Tridecane	1300	MS/KI	0.02	0.02	0.923	0.002
124	Tetradecane	1400	MS/KI	0.02	0.05	0.122	0.010
125	Pentadecane	1500	MS/KI	0.11	0.12	0.669	0.014
	<i>Aromatic hydrocarbons</i>			3.34	2.93	0.566	0.347
51	Methylbenzene (Toluene)	768	MS/KI	0.70	0.60	0.250	0.043
63	Ethylbenzene	865	MS/KI	0.30	0.29	0.918	0.050

Table 2 (continued)

Peak No.	Compounds	Feeding system					
		KI	Id	OUT	IND	P	SEM
65	1,3-Dimethylbenzene ( <i>m</i> -xylene)	873	MS/KI	1.64	1.32	0.430	0.196
69	1,4-Dimethylbenzene ( <i>p</i> -xylene)	898	MS/KI	0.36	0.32	0.514	0.030
80	1,2,4-Trimethylbenzene	976	MS/KI	0.06	0.05	0.372	0.007
86	1,2,3-Trimethylbenzene	988	MS/KI	0.04	0.06	0.670	0.021
91	1,3,5-Trimethylbenzene	1002	MS/KI	0.16	0.26	0.614	0.090
113	Naphthalene	1193	MS/KI	0.08	0.04	0.001	0.006
	<i>Chloride compounds</i>			1.42	2.54	0.082	0.322
21	Chloroform	615	MS/KI	1.42	2.54	0.082	0.322
	<i>Esters</i>			5.09	3.76	0.054	0.346
9	Methyl acetate	522	MS/KI	0.09	0.08	0.510	0.006
20	Ethyl acetate	612	MS/KI	3.15	2.25	0.052	0.233
35	Ethyl propanoate	714	MS/KI	0.13	0.11	0.541	0.013
37	Propyl acetate	717	MS/KI	0.14	0.16	0.581	0.016
49	Ethyl-2-methyl propanoate	760	MS/KI	0.07	0.08	0.464	0.010
55	Ethyl butanoate	796	MS/KI	0.65	0.39	0.073	0.073
56	Ethyl-2-hydroxy propanoate	809	MS	0.29	0.30	0.931	0.039
59	Ethyl-2-methyl butanoate	849	MS/KI	0.06	0.06	0.865	0.007
60	Ethyl-3-methyl butanoate	853	MS/KI	0.11	0.10	0.721	0.015
66	3-Methylbutyl acetate	877	MS/KI	0.03	0.02	0.166	0.003
89	Ethyl hexanoate	998	MS/KI	0.15	0.08	0.013	0.014
94	Hexylacetate	1011	MS/KI	0.03	0.03	0.875	0.003
112	Ethyl octanoate	1181	MS/KI	0.10	0.05	0.004	0.009
123	Ethyl decanoate	1395	MS/KI	0.08	0.05	0.059	0.009
	<i>Ethers</i>			0.12	0.35	0.217	0.092
7	1,1'-Oxybisethane (Ethyl ether)	505	MS/KI	0.12	0.35	0.217	0.092
	<i>Furans</i>			0.48	0.31	0.284	0.075
61	2-Furanmethanol (Furfuryl alcohol)	854	MS/KI	0.06	0.05	0.396	0.004
73	Dihydro-2(3H)-furanone (Butyrolactone)	917	MS/KI	0.15	0.17	0.339	0.011
77	Dihydro-5-methyl-2(3H)-furanone (Valerolactone)	958	MS/KI	0.01	0.01	0.431	0.001
87	2-Pentylfuran	994	MS/KI	0.05	0.04	0.190	0.004
101	5-Ethyl-dihydro-2(3H)-furanone	1057	MS/KI	0.20	0.04	0.266	0.070
122	5-Pentyl-dihydro-2(3H)-furanone (Nonalactone)	1378	MS/KI	0.01	0.01	0.000	0.001
	<i>Ketones</i>			11.29	16.34	0.215	2.009
5	2-Propanone + 2-propanol	<500	MS	0.81	3.97	0.044	0.792
17	2-Butanone	597	MS/KI	7.66	9.48	0.670	2.080
26	1-Hydroxy-2-propanone	663	MS/KI	0.09	0.09	0.706	0.007
30	2-Pentanone + propanoic acid	687	MS/KI	0.32	0.22	0.242	0.044
33	3-Hydroxy-2-butanone (Acetoin)	710	MS/KI	1.69	1.59	0.865	0.304
47	3-Methyl-2-pentanone	754	MS/KI	0.03	0.03	0.816	0.003
53	Cyclopentanone	787	MS/KI	0.50	0.85	0.056	0.092
68	2-Heptanone	891	MS/KI	0.13	0.07	0.002	0.011
85	3-Octanone	988	MS/KI	0.06	0.04	0.148	0.005
	<i>Nitrogen compounds</i>			0.66	0.63	0.763	0.044
39	3-Methylbutanenitrile	732	MS/KI	0.12	0.18	0.302	0.028
43	1-Methyl-1-H-pyrrole	744	MS/KI	0.11	0.13	0.458	0.014
45	Pyridine	747	MS/KI	0.08	0.08	0.768	0.006
48	1-(H)-pyrrole	756	MS/KI	0.01	0.01	0.639	0.000
72	2,6-Dimethylpirazine	915	MS/KI	0.07	0.06	0.214	0.004
93	Trimethylpyrazine	1006	MS/KI	0.03	0.02	0.280	0.002
106	Tetramethylpyrazine	1086	MS	0.26	0.16	0.004	0.017
	<i>Phenols</i>			0.30	0.23	0.040	0.018
81	Phenol	979	MS/KI	0.10	0.08	0.095	0.006
103	<i>p</i> -Cresol	1069	MS/KI	0.10	0.06	0.001	0.006
107	2-Methoxyphenol	1091	MS/KI	0.10	0.09	0.390	0.008
	<i>Sulphur compounds</i>			7.07	7.87	0.544	0.638
3	Methanethiol	<500	MS	0.13	0.20	0.068	0.020

Table 2 (continued)

Peak No.	Compounds	Feeding system					
		KI	Id	OUT	IND	P	SEM
8	Thiobis methane (Dimethyl sulfide)	517	MS/KI	0.04	0.09	0.007	0.008
10	Carbon disulfide	534	MS/KI	0.09	0.11	0.583	0.020
16	2-Propen-1-thiol	594	MS	1.07	0.57	0.130	0.162
31	(E)-3-(methylthio)-1-propene (Methylallylsulfide)	697	MS/KI	4.68	5.66	0.334	0.497
36	1-(methylthio)-propane	715	MS/KI	0.18	0.20	0.493	0.019
38	(Z)-1-(methylthio)-1-propene	725	MS/KI	0.13	0.16	0.198	0.012
41	(Z)-3-(methylthio)-1-propene	738	MS/KI	0.31	0.36	0.325	0.027
44	Dimethyl disulfide	747	MS/KI	0.03	0.12	0.080	0.026
62	3,3'-Thiobis-1-propene (Diallyl sulfide)	861	MS/KI	0.18	0.17	0.730	0.020
74	Methyl-2-propenyl disulfide (Methylallyldisulfide)	923	MS/KI	0.07	0.10	0.257	0.012
83	3-(methylthio)-1-propanol	981	MS	0.05	0.05	0.819	0.008
84	Dimethyl trisulfide	982	MS/KI	0.01	0.02	0.307	0.003
105	Di-2-propenyl disulfide (Diallyl disulfide)	1085	MS/KI	0.11	0.05	0.112	0.018
	<i>Terpenes</i>			3.05	2.61	0.581	0.384
76	Alpha-pinene	943	MS/KI	0.01	0.01	0.883	0.002
78	Camphene	961	MS/KI	0.02	0.02	0.816	0.002
95	Alpha terpinene	1018	MS/KI	0.03	0.03	0.682	0.003
97	<i>l</i> -Limonene	1036	MS/KI	0.64	1.00	0.512	0.275
99	1,8-Cineole (Eucaliptol)	1041	MS/KI	0.03	0.03	0.789	0.002
102	Gamma-terpinene	1063	MS/KI	0.04	0.04	0.925	0.004
104	Dihydro-myrcenol	1069	MS/KI	0.06	0.02	0.012	0.008
108	Linalool	1095	MS/KI	0.06	0.02	0.001	0.006
115	Trans-ocimene	1258	MS	0.01	0.01	0.878	0.001
117	Endobornyl acetate	1308	MS	1.86	1.30	0.150	0.193
119	Terpene	1364	MS	0.23	0.09	0.008	0.028
120	Terpene	1367	MS	0.07	0.05	0.257	0.006

OUT: dry-cured loins from pigs reared outdoors; IND: dry-cured loins from pigs fed on high oleic concentrate; Peak No.: Order of appearance of the peak within the identified peaks; KI: Kovats index; MS: Mass spectra; Id: Reliability of identification; SEM: Standard error of the mean.

molecular weight ones, like ethanol, 2-butanol and branched alcohols, most likely arise from microbial fermentation (Kandler, 1983; Sunesen, Dorigoni, Zanardi, & Stahnke, 2001). 3- and 2-methylbutanol come from reduction of 3- and 2-methylbutanal which in turn are derived from leucine and isoleucine degradation as result of Strecker reactions or microbial metabolism of these amino acids (Ruiz et al., 2002).

Aldehydes were about 7% of total chromatographic area, benzeneacetaldehyde being the major one. This compound arises from Strecker degradation of phenylalanine (Belitz & Grosch, 1997). Hexanal reached very low levels compared to results found in dry-cured ham (Ruiz et al., 2001). The lower levels of straight chain aldehydes such as hexanal, heptanal, octanal and nonanal suggest a lesser importance of lipid oxidation in dry-cured loin. 2-Methylpropanal (co-eluted with propanol), 2-methylbutanal (co-eluted with 1-butanol and benzene) and 3-methylbutanal are more likely formed as a consequence of Strecker degradation of the amino acids valine, isoleucine and leucine (Belitz & Grosch, 1997) or due to microbial metabolism (Hinrichsen & Pedersen, 1995). Aldehydes have a great impact on meat product aroma due to their low odour threshold values.

They exhibit characteristics aroma notes, such as butter, sweet, floral, toasted or green odours (Belitz & Grosch, 1997; Stahnke, 1998).

Aliphatic hydrocarbons were 4–8.5% of the total chromatographic area. Straight chain aliphatic hydrocarbons with less than 10 carbon atoms arise mainly from lipid oxidation (Ansorena, Gimeno, Astiasarán, & Bello, 2001; Ruiz et al., 2002), while those with longer chains could be accumulated in the fat depots of the animal, probably from feeding (Meynier, Novelli, Chizzolini, Zanardi, & Gandemer, 1999; Tejada, García, Petró, Andrés, & Antequera, 2001). Due to their high threshold values, the presence of these volatile compounds seems to have a limited influence on dry-cured product aroma (Ansorena et al., 2001).

Aromatic hydrocarbons reached 2.15–4.23% of the total chromatographic area, 1,3-dimethylbenzene (*m*-xylene) being the major one. This compound and 1,4-dimethylbenzene (*p*-xylene) could be stored in fat depots coming from animal feeding (Meynier et al., 1999). Methylbenzene (toluene) possibly derived from cyclation of unsaturated carboxylic chains produced by lipid degradation (Min, Ina, Peterson, & Chang, 1977) or from ambient contamination. Naphthalene has been

cited among the volatile compounds of smoked paprika (Mateo et al., 1997), thus it could arise from paprika utilized in the pickling sauce.

A variety of esters, mainly ethyl esters were found, though quantitatively they were not very important (3–5% of the total chromatographic area). The major ester found was ethyl acetate, which has been described as the main ester in different types of dry-cured sausages (Bruna et al., 2001; Schmidt & Berger, 1998). Ethyl esters are formed through esterification reactions between ethanol and carboxylic acids (Peterson & Chang, 1982). Some authors have studied the ability of microorganisms to form these compounds (Stahnke, 1994). Esters found in Iberian dry-cured loins have been described previously in different dry-cured sausages and in Parma ham. In these products, the relevance of microorganisms in the formation of volatile compounds has been proved, (Bruna et al., 2001; Hinrichsen & Pedersen, 1995). Esters from short chain fatty acids, acetates, propanoates and butanoates, exhibit fruity notes and low odour thresholds and thus can contribute to dry-cured sausage aroma (Stahnke, 1995b).

Furans constituted 0.30–0.57% of the total volatile compounds identify. The main furan identified was dihydro-2-(3H)-furanone (butyrolactone). These types of compounds in foods are related with the heating processes. However, they are commonly described among volatiles of dry-cured ham and dry-cured sausages, and their formation has been demonstrated in model systems of linoleic acid (C18:2,  $n - 6$ ) at 20 °C (Grosch, 1987). Furfuryl alcohol could arise from paprika (Mateo et al., 1997). 2-Pentylfuran, is a non-carboxylic compound derived from linoleic acid (C18:2,  $n - 6$ ) and other  $n - 6$  fatty acids (Frankel, 1991) and has a low odour threshold (Forss, 1988). This compound has been found in Iberian dry-cured ham and other types of meat products (Andrés et al., 2002; Dirinck, Van Opstaele, & Vandenriessche, 1997). Ruiz et al. (2002) attribute the presence of lactones in dry-cured Iberian ham to lipid oxidation reactions, though Maillard reactions should not be ignored. In dry-cured loins the addition of smoked ingredients such as paprika, could contribute to the presence of lactones (Mateo & Zumalacárregui, 1996). This kind of compound shows characteristic pleasant odours, defined as cocoa, butter or fruity (Belitz & Grosch, 1997) and low odour thresholds. This type of compound determines the characteristic odour of French fries (Wagner & Grosch, 1998) and roasted beef (Cerny & Grosch, 1992) and they are thought to be important in dry-cured ham aroma (Ruiz et al., 2002).

Ketones represented 11–16% of total volatiles, among them 2-butanone showed the highest percentage. Straight chain ketones can derive from lipid oxidation (Belitz & Grosch, 1997), while 1-hydroxy-2-propanone, 3-hydroxy-2-butanone or acetoin and 3-methyl-2-pentanone arise from microbial fermentation of glucides

(Kandler, 1983). Production of 2-butanone and other “methyl-ketones” has also been associated with  $\beta$ -oxidation activity of moulds growing in the surface of dry-cured products, as *Penicillium nalgiovense* (Sunesen & Stahnke, 2003), which in turn, has been found among the microbial population of the surface of Iberian dry-cured ham (Núñez et al., 1996). Ketones, especially 2-ketones, are considered to have a great influence on the aroma of cheese, meat and meat products as they are present in large amounts and have characteristic aromas, such as ethereal, butter, spicy notes or blue cheese notes (Lecanu, Ducruet, Jouquand, Gratadoux, & Feigenbaum, 2002; Novelli, Gandemer, Meynier, Zanardi, & Chizzolini, 1995).

Nitrogen compounds represented 0.6% of total chromatographic area. The higher levels corresponded to tetramethylpyrazine. Nitrogen compounds in dry-cured meat products come principally from Maillard reactions between lipid oxidation products and certain amino acids (Ruiz et al., 2002) although paprika might be a source of nitrogen compounds (Mateo et al., 1997). Pirazines and pyrrols derived from Maillard reactions are commonly found in dry-cured products (Andrés et al., 2002; Schmidt & Berger, 1998). Pirazines have characteristic walnut, toasted or potato odours (Meynier et al., 1999). Nitriles could arise from reactions between aldehydes and curing agents (Mottram, 1984).

Only three phenolic compounds were found, representing 0.23–0.30% of total chromatographic area. These types of compound may derive from pyrolysis of lignine and exhibit low odour thresholds with characteristic smoke aromas (Stahnke, 1998). In dry-cured sausages the presence of a wide range of phenolic compounds has been reported (Ansorena et al., 2001). In dry-cured loin, phenols could come from paprika (Mateo et al., 1997). However, Ruiz, Ventanas, Cava, Andrés, and García (1999), identified 3-methylphenol among Iberian dry-cured ham volatiles though this is a non-smoked and non-spiced product.

Sulphur compounds were 8% of total chromatographic area. (E)-3-(methylthio)-propene showed the highest levels. The presence of some of the sulphur compounds, such as methanethiol, carbon disulfide, dimethyl disulfide or trimethyl trisulfide have been described as the result of reaction between the sulphur amino acids, cysteine and methionine, and carboxylic compounds from lipid oxidation like 2,3-pentadione (revised by Ruiz et al., 2002). However, garlic used in the pickling sauce is the main source of the compounds found. Thus, the major one, (E)-3-(methylthio)-propene has been described by Mateo and Zumalacárregui (1996) among volatile compounds in garlic. Many of the sulphur compounds found in this study have been previously found in dry-cured sausages in which garlic is an ingredient. This is the case of 2-propen-1-thiol, 3,3'-(thio-bis)-propene (allyl disulfide), 1-(methylthio)-propane,

(Z)-1-(methylthio)-propene and methyl-2-propenyl disulfide (methyl allyl disulfide) (Mateo & Zumalacárrgui, 1996; Sunesen et al., 2001). Some of these compounds probably arise from the complex reactions that the garlic sulphur compounds, derived from allicin, undergo when brought into contact with protein, salt and water (Bocchini, Andaló, Pozzi, Galletti, & Antonelli, 2001; Sunesen et al., 2001). Sulphur compounds possess intense odours described as sulphuric, cauliflower or onion (Acree & Arn, 1997; Meynier et al., 1999). Because of their characteristic low odour thresholds (Mottram, 1991) these types of compounds could determine the aroma of the products, over-riding the aromatic notes of other volatile compounds and giving the product its characteristic aroma (Edwards, Ordóñez, Dainty, Hierro, & De la Hoz, 1999). Thus, Stahnke (1998) identified 2-propen-1-thiol (allyl-1-thiol) and methylthiirane (detected in trace amounts in the loins) as having characteristic salami aroma.

Terpenes were 1.89–3.52% of total volatiles. Endobornyl acetate and L-limonene were the main terpenes present. Terpenes have two possible origins, accumulation in fat depots from animal feeding or spices added during processing. In spiced dry-cured products, terpenes have quantitatively and qualitatively great relevance. Meynier et al. (1999) found that in Milano salami 50% of the total volatile compounds were terpenes, due to the utilization of black pepper as an ingredient. Viallon et al. (1996) in dry-cured sausages seasoned with pepper found 12 different terpenes. Characteristic aromas of terpenes are well established, thus, alpha-pinene has a typical pine odour, camphene a camphor odour, alpha-terpinene and limonene exhibit lemon notes while linalool possesses flowery notes (Acree & Arn, 1997; Belitz & Grosch, 1997). However, olfactometric techniques applied by Croizet et al. (1992) in French dry-cured sausages, showed that most terpenes have a very limited role in the intensity and variety of aroma. Schmidt and Berger (1998) concluded that only linalool contributes markedly to the final aroma of dry-cured products, though terpenes were the major fraction of the total volatiles identified. Meynier et al. (1999) found only 7 terpenes with any aromatic note in a total of 26 terpenes found in Milano salami.

### 3.2. Effect of pig genetic line and feeding

No significant effect of Iberian pig line on the volatile profile of Iberian dry-cured loin was found. Therefore, results showing the individual proportion of each volatile compound of each line have not been shown.

Feeding systems influenced the levels of various compounds (Table 2). Dry-cured loins from OUT pigs had lower levels of 2-methylpropanoic acid ( $P = 0.020$ ) and higher levels of octanoic ( $P = 0.000$ ), nonanoic ( $P = 0.000$ ) and decanoic acid ( $P = 0.001$ ).

Among the alcohols, 1-methoxy-2-propanol ( $P = 0.024$ ), cyclohexanol ( $P = 0.003$ ), 2-butoxyethanol ( $P = 0.004$ ) and the co-elution formed by 1-butanol, 2-methylbutanal and benzene ( $P = 0.000$ ) reached higher levels in loins from animals fed concentrated feed, while 1-octen-3-ol ( $P = 0.000$ ) was 2-fold higher in OUT batch loins. This compound comes from linoleic acid (C18:2,  $n - 6$ ) oxidation (Whitfield & Last, 1991) which in a previous paper was at significantly higher levels in fresh meat samples from the same animals (Muriel et al., 2002). 1-Octen-3-ol has been repeatedly identified as having a marked mushroom odour and very low odour threshold (Meynier et al., 1999; Novelli et al., 1995).

The total aldehydes percentage of the chromatographic area ( $P = 0.001$ ) and individual levels of hexanal ( $P = 0.007$ ), heptanal ( $P = 0.036$ ), octanal, ( $P = 0.001$ ), nonanal ( $P = 0.000$ ) and 2-methylundecanal ( $P = 0.001$ ) were significantly higher in the OUT group. These results agree with those reported by López et al. (1992) who found higher levels of these aldehydes in dry-cured Iberian hams from pigs fed outdoors. Hexanal is the main compound derived from the oxidation of  $n - 6$  fatty acids, heptanal arises from oleic (C18:1,  $n - 9$ ) or linoleic (C18:2,  $n - 6$ ) autooxidation and finally, octanal and nonanal comes from oleic (C18:1,  $n - 9$ ) acid oxidation. Muriel et al. (2002), found similar levels of oleic acid (C18:1,  $n - 9$ ) in the fresh muscles used to produce the present dry-cured loins. However, levels of fatty acids belonging to  $n - 3$  family and linoleic acid (C18:2,  $n - 6$ ) were significantly higher in loins from the OUT pig group. The higher levels of polyunsaturated fatty acids, especially eicosapentaenoic acid (C20:5,  $n - 3$ ) (EPA) and docosahexanoic acid (C22:6,  $n - 3$ ) (DHA) in loins from OUT pigs, could make them more prone to lipid oxidation.

2-Methylpropene ( $P = 0.029$ ) and pentane ( $P = 0.040$ ) were at higher levels in IND pigs while decane ( $P = 0.030$ ) was 15-fold higher in dry-cured loins from OUT pigs. On the other hand, among aromatic hydrocarbons, only naphthalene showed differences between the batches, with higher levels in those loins from OUT pigs. This compound has been found in the volatile compounds of Spanish paprika though the differences suggest its accumulation in fat depots from grass ingested by animals during the fattening period was mainly responsible.

Ethyl hexanoate ( $P = 0.013$ ) and ethyl octanoate ( $P = 0.004$ ) were higher in loins from OUT pigs and the differences found in the latter compound agree with the high levels of octanoic acid in these samples. However, hexanoic acid co-eluted with benzaldehyde and so, such relationship could not be established.

Regarding ketones, co-elution by 2-propanone and 2-propanol was significantly higher in those loins from IND pigs ( $P = 0.044$ ). On the other hand, 2-heptanone was higher in loins from the OUT batch ( $P = 0.002$ ).

This ketone has been detected in dry-cured ham and dry-cured sausages (Ansorena et al., 2001; Carrapiso et al., 2002; Edwards et al., 1999; Meynier et al., 1999) and has been postulated as a key compound in dry-cured Iberian ham aroma (Carrapiso et al., 2002).

Terpenes showed higher amounts of dihydromyrcenol and linalool in loins from OUT pigs ( $P = 0.012$  and  $P = 0.001$ , respectively) which could be related to the higher accumulation of terpenes from feeds in the adipose tissues of OUT animals than in IND ones.

Among other families, only dimethyl sulphide exhibited higher percentages ( $P = 0.007$ ) in dry-cured loins from IND pigs, while compounds such as 5-pent-ildihydro-2(3H)-furanone (nonalactone) ( $P = 0.000$ ) were more abundant in loins from OUT pigs.

#### 4. Conclusions

Feeding regime affects the volatile profiles of dry-cured loin, mainly the aldehydes derived from oxidation of oleic acid (C18:1,  $n - 9$ ) and linoleic acid (C18:2,  $n - 6$ ) and compounds most likely coming from acorn and grass, such as decane, naphthalene, dihydromyrcenol and linalool. On the other hand, no difference in volatile profile was found relating to genetic line.

The results showed that in Iberian dry-cured loin there exists a predominance of those volatile compounds coming from microbial metabolism or from spices, such as ethanol, acetic acid, 2-butanone and numerous ethyl esters and sulphur compounds. On the contrary, compounds derived from lipid oxidation, mainly aldehydes, were of low relative importance.

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