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Volatile Profiles of Dry-Cured Meat Products from Three Different Iberian X Duroc Genotypes

ROSARIO RAMÍREZ AND RAMÓN CAVA*

Tecnología de los Alimentos, Facultad de Veterinaria, Universidad de Extremadura, Cáceres 10071, Spain

Volatile profiles of two Iberian dry-cured products, dry-cured loin and ham, from three different Iberian × Duroc genotypes, was assessed. Three groups of 10 pigs, each (5 males and 5 females) from different genotypes, were studied: GEN1 = ♂ Iberian × ♀ Duroc1; GEN2 = ♂ Duroc1 × ♀ Iberian; and GEN3 = ♂ Duroc2 × ♀ Iberian. The genotype Duroc1 (DU1) corresponded to pigs selected for the production of dry-cured meat products (hams, loins, and shoulders), with a high level of fattening, while the genotype Duroc2 (DU2) corresponded to animals selected for meat production. Genotype slightly affected the volatile profiles of both dry-cured meat products, although dry-cured loin from GEN3 showed higher hexanal content. Dry-cured loin showed a volatile profile very different to that found in dry-cured ham. Volatile compounds of dry-cured meat products were mainly originated by lipid and protein degradation. Most of the volatile detected in both meat products came from lipid oxidation such as acids, aldehydes, ketones, alcohols, and hydrocarbons. In addition, a high proportion of volatile compounds from the Maillard reaction was found. Branched aldehydes and some sulfur and nitrogen compounds have their origin in the amino acids degradation by the Strecker reaction, while branched alcohols and acids come from the lipid oxidation of branched aldehydes. Dry-cured ham showed a higher number and a higher level of compounds with origin in protein and lipid degradation than dry-cured loin, which agrees with the longer ripening of the hams (24 months) with respect to the loins (4 months). In dry-cured loins, apart from these compounds, seasoning mixture provides high amount of volatiles, such as terpenes (from paprika and oregano) and sulfur compounds (from garlic), which have great importance in the overall aroma of this product.

KEYWORDS: Volatile; SPME; Duroc; Iberian; dry-cured loin; dry-cured ham.

INTRODUCTION

Iberian dry-cured loin and ham are the most valuable dry-cured meat products, with an extraordinary consumer acceptance because of their sensory quality, especially their unique and characteristic flavor. The main factors that impact the characteristic and intense flavor of these products are the meat quality as well as the special features of the ripening process, such as its length (14−36 months for dry-cured hams and 3−6 months for dry-cured loins).

The quality of the dry-cured meat products is closely related to the characteristics of the raw material, especially those related to the degree of marbling and the fatty acid composition of fat, such as feeding characteristics, age of animals, and pig breed (*1*). One of the alternatives more often applied to improve productive parameters of Iberian pig is by the cross with Duroc breed (*2*). However, different studies have demonstrated that differences between Duroc lines affect productive characteristics and meat and meat products quality (*3*). Therefore, the Duroc line could affect the aroma of Iberian dry-cured meat products.

The manufacture process and ripening of loins and hams influence their flavor. Iberian dry-cured loin is manufactured by rubbing a mixture of curing agents (salt and nitrite) and spices (i.e., Spanish paprika, oregano, and garlic) on the surface of the loins. Then, they are stuffed into casings and subsequently ripened. Previous researchers have established that the spices rubbed onto the surface play an important role in the flavor of dry-cured loin (*4*,*5*). Moreover, the antioxidant effect of the spices and nitrites added to dry-cured sausages has been probed by Aguirrezábal et al. (*6*), and it could affect the development of lipid oxidation reactions. Nonetheless, dry-cured hams are only covered with salt and nitrites and undergo a much longer ripening process; therefore, greater development of lipid and protein degradation reactions would be expected.

A considerable amount of studies have been devoted to describe the volatile flavor compounds of Iberian hams (i.e., *7*, *8*), while there are a few previous studies about Iberian loin flavor (*4*, *5*). In addition, there are several works about reactions that provide volatile compounds such as lipolysis, lipid oxidation, Maillard reaction and protein and amino acids degradation, and the contribution of microorganisms to the flavor development in Iberian products. However, the comparison between

* Phone: +34 927 257 169. Fax: +34 927 257 110. E-mail: rcava@unex.es.

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the volatile profile of Iberian dry-cured loin and dry-cured ham has never been carried out.

Lipid derived compounds are the most abundant compounds in dry-cured meat products (*9*,*10*). The extent of lipolysis and lipid oxidation determines the final flavor of dry-cured meat products (*11*). On the other hand, the Maillard reaction, which occurs between amino compounds and reducing sugars, is one of the most important routes of flavor compounds in dry-cured products (*12*). Besides, compounds from the Maillard reaction can also react with other components of meat such as aldehydes and other carbonyls formed during lipid oxidation, which react readily with Maillard intermediates. Such interactions contribute to the achievement of the optimum and characteristic flavor of meat products (*13*).

Therefore, the main objectives of this paper are the characterization of volatile compounds of dry-cured loin and ham and to determine the effect of the genotype on the volatile compounds profile extracted from dry-cured meat products from different Iberian × Duroc genotypes.

MATERIALS AND METHODS

Animals. Three groups of 10 pigs each one (5 males and 5 females) from different genotypes were studied: GEN1 = ♂ Iberian × ♀ Duroc1; GEN2 = ♂ Duroc1 × ♀ Iberian; and GEN3 = ♂ Duroc2 × ♀ Iberian. The genotype Duroc1 (DU1) corresponded to pigs selected for the production of dry-cured meat products (hams, loins, and shoulders), with a high level of fattening. The genotype Duroc2 (DU2) corresponded to animals selected for meat production, with high percentages of meat cuts and with low carcass fattening. Pigs were intensively raised all together and fed *ad libitum* with a cereal-based commercial fodder. Pigs were randomly slaughtered after 316 days of rearing with 150−165 kg of live weight.

Dry-Curing Process. For the manufacture of the dry-cured loins, the *Longissimus dorsi* muscle (10 loins/genotype) was removed from the carcass and processed into dry-cured loin. The weight of the fresh muscle was 2.5−3.2 kg, and pH values were lower than 6.0. Loins were seasoned by rubbing a mixture of salt, nitrite, olive oil, and spices such as Spanish paprika (*Capsicum annuum, L.*), oregano (*Origanum vulgare, L.*), and garlic (*Altium sativum, L.*). Loins were kept for 4 days at 4 °C to allow the seasoning mixture to penetrate. Then, loins were stuffed into collagen casings and held for 30 days at 4 °C at a relative humidity of ∼80%. Finally, loins were ripened for 90 days at ∼12 °C and at ∼70% relative humidity. Loins were processed for a total dry-curing time of 4 months. For the analysis of volatile compounds in this product, the surface (3 mm) with the pickling mixture was removed.

For the manufacture of dry-cured hams, one ham from each animal was processed (10 hams/genotype). The weight of the fresh ham was 14.0−15.5 kg, and pH values were <6.0. After salting, hams were kept at 0−3 °C and 80−90% relative humidity for ∼6 months. Then, the hams were ripened for ∼18 months at 10−25 °C and 60−80% relative humidity. Hams were processed for a total dry-curing time of 24 months. After the ripening process, the *Biceps femoris* muscles were removed from the hams for the analysis.

Volatile Analyses. A SPME fiber (Supelco Co., Canada) coated with divinylbenzene−carboxen−poly(dimethylsiloxane) (DVB/CAR/PDMS) 50/30 μm was used. The sampling technique to extract volatile compounds from the headspace was the following: 0.5 g of meat were minced and placed in 5 mL vials with a silicone stopper, which were previously deodorized by heating them in an electric stove at 80 °C for at least 2 h. Each sample was analyzed in duplicate. The vial was maintained in a temperature-controlled water bath at 37 °C. The fiber was exposed to the headspace of the sample for 30 min. The analyses were performed on a HP5890GC series II gas chromatograph (Hewlett-Packard) coupled to a mass-selective detector, Agilent model 5973. Volatiles were separated using a 5% phenyl−95% dimethylpolysiloxane column (30 m × 0.25 mm i.d., 1.0 mm film thickness; Restek). The carrier gas was helium at 18.5 psi, resulting in a flow of 1.6 mL/min at 40 °C. Prior to the analysis, the SPME fiber was preconditioned at 270 °C for 50 min in the gas chromatography injection port. The injection port was in *splitless* mode, and the temperature program was isothermal at 40 °C for 10 min and then raised at the rate of 7 °C seg^{-1} to 250 °C and held for 5 min. The GC-MS transfer line temperature was 270 °C. The MS operated in the electron impact mode with an electron impact energy of 70 eV, with a multiplier voltage of 1650 V and a collected data rate of 1 scan s^{-1} over a range of *m*/*z* 40−300. *n*-Alkanes (Sigma R-8769) were analyzed under the same conditions to calculate the retention indices (RI) for the volatiles. The compounds were identified (i) by comparison with commercial reference compounds (Sigma-Aldrich), (ii) by comparison of RI with those described by Kondjoyan and Berdagué (*14*) and on the web at http://webbook.nist.gov/, and (iii) by comparison of their mass spectra with those contained in the Wiley library.

Statistical Analysis. The effects of genotype and sex were analyzed using the analysis of variance (ANOVA) procedure of SPSS, version 12.0. A two-way analysis of the variance (genotype and sex) with interaction (genotype × sex) was carried out. Because genotype × sex was not significant, this data is not show in the table. HSD Tukey's test was applied to compare the mean values of the genotypes. Mean values and standard errors of the means (SEM) are reported. The relationship between parameters was assessed by the principal component analysis (PCA).

RESULTS AND DISCUSSION

Origin of the Volatile Compounds of Iberian Dry-Cured Meat Products. Fourty-one compounds were isolated in dry-cured loin (**Table 1**): 5 acids, 4 ketones, 3 alcohols, 11 aldehydes, 3 esters, 2 nitrogen compound, 3 sulfur compounds, 3 terpenes, 4 lineal hydrocarbons, and 3 aromatic hydrocarbons. In dry-cured ham (**Table 2**), a higher number of volatiles than in dry-cured loin was detected (55 compounds): 7 acids, 11 ketones, 8 alcohols, 12 aldehyes, 7 lineal hydrocarbons, 2 esters, 3 nitrogen compounds, 3 sulfur compounds, 1 terpene, and 1 aromatic hydrocarbon. The most abundant compounds in dry-cured loin were aldehydes (45.7%), followed by terpenes (17.0%) and ketones (10.1%). Similarly, aldehydes (47.7%) had the highest chromatographic area in dry-cured ham, followed by alcohols (24.7%) and ketones (16.7%).

The relative amount of volatile compounds was twice higher in dry-cured ham than in dry-cured loin (**Figure 1**), since the volatile profile of dry-cured ham showed a larger amount of lipid derived compounds (aldehydes, ketones, and alcohols) and Maillard compounds than that of dry-cured loin. In general, differences in the volatile profilse of dry-cured hams and loins are attributable to the different manufacturing process and ripening length. So, the shorter ripening process (4 months vs 24 months) and the lower temperatures during the dry-cured loin processing compared to that of dry-cured hams would probably contribute to a more limited development of the chemical reactions involved in flavor compounds generation in dry-cured loin. As a consequence, the formation of lipid and

Table 1. Volatile Compounds (area units (AU) × 10^6) Detected in the Headspace of Dry-Cured Loins from Three Different Iberian × Duroc Genotypes

			genotype[c]			sex		probabilities		
RI[a]	id. method[b]		GEN1	GEN2	GEN3	♂	♀	SEM[d]	gen	sex
		Lipid Oxidation (55.8%)								
		Acids								
970	RI,TI	hexanoic acid	11.2	12.7	16.0	14.5	12.3	1.8	0.615	0.595
		Ketones								
502	RI,TI	propan-2-one	17.2	27.9	34.0	24.8	28.5	8.4	0.727	0.795
891	RF,RI,TI	heptan-2-one	2.0	2.2	3.8	3.0	2.4	0.5	0.216	0.574
983	RI,TI	octane-2,3-dione	14.3 b	28.0 ab	55.9 a	31.7	34.9	5.6	0.008	0.651
		Alcohols								
868	RF,RI,TI	hexan-1-ol	2.3	2.9	3.1	2.6	2.9	0.2	0.366	0.580
900	RI,TI	heptan-2-ol	6.2	5.2	6.4	6.5	5.3	0.5	0.584	0.266
		Aldehydes								
696	RI,TI	pentanal	4.4	8.5	10.1	6.9	8.6	1.1	0.104	0.392
800	RF,RI,TI	hexanal	201.0 b	307.9 b	651.2 a	384.8	400.8	61.3	0.006	0.767
901	RF,RI,TI	heptanal	7.1b	7.1b	14.7 a	11.2	8.4	1.2	0.006	0.206
956	RF,RI,TI	(*E*)-hepten-2-al	0.6	2.4	1.8	1.9	1.4	0.4	0.204	0.600
1004	RF,RI,TI	octanal	4.7	3.6	4.9	4.7	4.2	0.6	0.676	0.681
1107	RI,TI	nonanal	17.4	14.7	14.2	15.9	14.9	1.1	0.493	0.614
1208	RI,TI	decanal	0.9	0.3	0.4	0.9	0.2	0.2	0.413	0.142
		Lineal Hydrocarbons								
556	TI	2-methylpentane	1.8 ab	3.1 a	1.2 b	2.3	1.8	0.3	0.039	0.449
576	TI	3-methylpentane	1.8	7.9	1.5	2.3	5.2	1.7	0.229	0.397
699	RF,RI,TI	heptane	4.3	5.1	3.4	4.1	4.4	0.4	0.100	0.730
1002	RF,RI,TI	decane	1.2	2.1	1.5	1.9	1.4	0.3	0.546	0.491
		Aromatic Hydrocarbons								
	TI	methylbenzene	15.1	44.7	50.8	49.8	26.3	11.5	0.458	0.382
878	RI, TI	1,3-dimethylbenzene	17.2	13.9	14.8	15.5	15.0	0.8	0.258	0.697
976	TI	1,2,3-trimethylbenzene	1.3	1.9	1.1	1.6	1.4	0.4	0.745	0.863
		Maillard Reaction (18.1%)								
		Acids								
	TI	2-methylpropanoic acid	3.7	4.2	3.9	4.9	3.1	1.0	0.990	0.397
839	RI,TI	2-methylbutanoic acid	18.9	21.2	19.1	23.2	16.6	3.3	0.969	0.333
843	RI,TI	3-methylbutanoic acid	6.2	3.8	4.3	5.7	3.9	0.9	0.455	0.279
		Alcohols								
	TI	propan-2-ol	46.8	98.2	43.1	69.3	57.6	14.3	0.222	0.714
		Aldehydes								
	RF, TI	acetaldehyde	40.2	6.9	11.3	6.5	30.2	10.8	0.511	0.276
647	RF,RI,TI	3-methylbutanal	21.7	23.1	15.8	15.3	24.7	2.7	0.488	0.098
657	RF,RI,TI	2-methylbutanal	3.2	1.0	2.5	1.0	3.3	1.0	0.736	0.282
1055	RI,TI	benzene acetaldehyde	3.5	3.1	1.7	2.3	3.1	0.6	0.434	0.614
		Ketones								
712	RI,TI	3-hydroxybutan-2-one	78.6	27.5	20.2	67.7	15.8	23.8	0.469	0.229
		Nitrogen Compounds								
915	RI,TI	2,6-dimethylpyrazina	5.0	4.1	4.5	4.5	4.5	0.4	0.701	0.911
	TI	ethamina-*N,N*-diethyl	5.0	3.1	3.7	5.0	2.9	0.9	0.682	0.253
		Microorganisms Esterification (0.8%)								
856	RI,TI	butanoic acid 3-methylethyl ester	3.2	4.2	3.2	4.1	3.1	0.4	0.424	0.210
1034	TI	hexanoic acid 1-methylethyl ester	4.0	4.4	4.3	4.4	4.1	0.4	0.948	0.761
1195	TI	octanoic acid methyl ester	0.2	1.3	0.5	0.8	0.6	0.2	0.083	0.762
		Species (25.2%)								
		Acids								
615	TI	acetic acid	19.7	10.7	11.1	15.8	11.6	3.9	0.546	0.533
		Sulfur Compounds								
864	TI	prop-1-ene-3,3'-thiobis	17.0	15.4	12.3	14.9	14.8	1.6	0.520	0.921
923	RI,TI	2-propenylmethyldisulfide	3.5	3.9	4.0	4.1	3.5	0.3	0.850	0.316
1085	RI,TI	dipropenyldisulfide (diallyl disulfide)	51.8	57.9	44.1	48.3	54.0	3.2	0.182	0.418
		Terpenes								
935	RI,TI	alpha-pinene	106.3	98.6	76.2	93.7	92.9	7.6	0.293	0.913
1014	RI,TI	delta-3-carene	79.8	76.6	68.2	78.2	71.5	7.6	0.823	0.658
1039	RI,TI	1-limonene	7.8	6.8	6.2	7.7	6.1	0.7	0.528	0.198

[a] RI = retention index. [b] Method of identification: RF, mass spectrum and retention time identical with a reference compound; RI, mass spectrum and retention index from literature in accordance; TI, tentative identification by mass spectrum. [c] GEN1 = IB × DU1; GEN2 = DU1 × IB; GEN3 = DU2 × IB. Different letters in the same row (a, b) indicate significant statistical differences (Tukey's Test, $p < 0.05$). [d] SEM = standard error of the mean.

Table 2. Volatile Compounds (area units (AU) × 10^6) Detected in the Headspace of Dry-Cured Hams from Three Different Iberian × Duroc Genotypes

			genotype[c]			sex		probabilities		
RI[a]	id. method[b]		GEN1	GEN2	GEN3	♂	♀	SEM[d]	gen	sex
		Lipid Oxidation (81.6%)								
		Acids								
779	RI,TI	butanoic acid	13.76	9.35	11.38	11.80	11.20	1.59	0.503	0.813
970	RI,TI	hexanoic acid	36.89	24.56	26.12	27.40	31.22	3.39	0.333	0.695
1179	RI,TI	octanoic acid	0.53	1.03	1.95	1.42	0.86	0.66	0.686	0.647
		Ketones								
502	RI,TI	propan-2-one	385.50	388.00	345.92	380.75	367.63	33.47	0.903	0.785
600	RF,RI,TI	butan-2-one	11.18	8.60	33.92	11.86	22.70	5.89	0.194	0.357
685	RF,RI,TI	pentan-2-one	39.15	32.74	31.54	36.14	33.04	4.11	0.772	0.624
792	RI,TI	hexan-2-one	8.83	7.26	3.70	6.25	7.16	1.15	0.228	0.762
891	RF,RI,TI	heptan-2-one	37.44	22.75	22.12	24.81	30.47	3.40	0.155	0.511
983	RI,TI	octane-2,3-dione	19.62	14.77	17.18	16.54	17.84	3.53	0.846	0.872
991	RI,TI	octan-2-one	1.80	0.00	0.00	0.33	0.92	0.42	0.172	0.605
1068	TI	1-etoxi-heptan-2-one	2.20a	0.00b	0.00b	0.66	0.86	0.34	0.008	0.959
1088	TI	non-8-en-2-one	1.80	0.00	0.00	0.38	0.87	0.45	0.228	0.717
1093	RI,TI	nonan-2-one	10.40	0.00	3.15	2.50	6.64	2.53	0.291	0.532
		Alcohols								
482	TI	ethanol	70.32	174.53	83.77	117.13	103.93	24.75	0.220	0.920
680	RI,TI	pent-1-en-3-ol	3.90	3.26	4.47	4.05	3.65	0.43	0.496	0.622
772	RF,RI,TI	pentan-1-ol	32.29	19.34	27.82	26.98	25.88	2.43	0.093	0.680
868	RF,RI,TI	hexan-1-ol	70.44 a	24.84 b	34.72 b	43.92	43.40	7.78	0.044	0.779
900	RI,TI	heptan-2-ol	1.64	2.31	1.51	1.95	1.72	0.33	0.552	0.743
982	RI,TI	oct-1-en-3-ol	10.90	6.31	10.77	9.11	9.44	1.14	0.206	0.997
		Aldehydes								
696	RI,TI	pentanal	51.00	46.20	54.89	47.39	53.68	5.71	0.871	0.635
800	RF,RI,TI	hexanal	646.60	563.29	809.43	641.47	694.26	48.65	0.160	0.635
901	RF,RI,TI	heptanal	56.05	41.74	49.26	49.48	48.53	5.03	0.519	0.871
956	RF,RI,TI	(E)-hepten-2-al	4.92 a	1.65 b	2.65 ab	3.24	2.94	0.49	0.012	0.560
1004	RF,RI,TI	octanal	46.26 a	23.40 b	31.24 ab	36.56	30.90	3.85	0.039	0.325
1107	RI,TI	nonanal	61.94	39.56	44.51	53.08	44.59	4.96	0.151	0.307
1208	RI,TI	decanal	1.17	0.30	0.00	0.51	0.51	0.22	0.094	0.878
		Lineal Hydrocarbons								
560	TI	2-methylpentane	0.97	1.75	1.28	1.59	1.09	0.46	0.685	0.547
578	TI	3-methylpentane	0.37	0.61	1.12	1.13	0.24	0.32	0.671	0.166
699	RF,RI,TI	heptane	7.69	2.02	9.36	6.84	5.64	1.50	0.093	0.499
1002	RF,RI,TI	decane	0.00	5.90	1.14	1.94	2.85	1.55	0.247	0.697
1215	TI	2,6-dimethylundecane	1.43	1.20	0.00	0.88	0.94	0.32	0.165	0.957
	TI	4-methyldodecane	2.64	2.86	0.00	2.30	1.51	0.56	0.098	0.501
	TI	2,6-dimethylnonane	15.37	9.95	6.26	11.04	10.35	1.68	0.092	0.776
		Aromatic Hydrocarbons								
878	RI,TI	1,3-dimethylbenzene	2.00	3.51	7.33	4.31	4.01	1.13	0.175	0.987
		Maillard Reaction (12.7%)								
		Acids								
758	RI,TI	2-methylpropanoic acid	5.97	5.35	10.80	8.38	6.10	1.42	0.289	0.415
839	RI,TI	2-methylbutanoic acid	8.15	11.30	26.27	17.40	12.23	3.42	0.088	0.435
843	RI,TI	3-methylbutanoic acid	1.29 b	0.00 b	12.56 a	6.29	2.33	2.05	0.021	0.243
		Ketones								
712	RI,TI	3-hydroxybutan-2-one	1.21	3.08	0.45	2.04	1.21	0.56	0.168	0.517
		Alcohols								
	TI	propan-2-ol	0.38	30.02	53.49	18.92	35.04	13.10	0.278	0.441
739	RI,TI	3-methylbutan-1-ol	18.13	6.94	10.24	15.72	7.94	3.87	0.420	0.288
		Aldehydes								
553	TI	2-methylpropanal	10.16	11.07	19.73	12.25	14.59	2.52	0.301	0.623
647	RF,RI,TI	3-methylbutanal	126.05	72.67	99.12	111.72	86.84	18.00	0.424	0.451
657	RF,RI,TI	2-methylbutanal	30.67	30.15	37.29	31.48	33.57	2.44	0.511	0.688
1055	RI,TI	benzene acetaldehyde	8.77	22.92	6.84	18.94	7.21	5.54	0.539	0.375
910	TI	3-methylthiopropanal	1.95	4.16	10.18	1.50	8.99	2.76	0.488	0.167
		Nitrogen Compounds								
751	RI,TI	pyridine	0.63	4.75	1.24	0.40	4.09	1.29	0.231	0.113
913	RI,TI	2,6-dimethylpyrazine	1.47	3.84	0.00	2.08	1.60	0.66	0.081	0.821
1010	TI	trimethylpyrazine	0.00	9.70	1.19	0.96	6.49	2.42	0.097	0.174
		Sulfur Compounds								
920	RI,TI	dihydro-2(3h)-furanone	1.76	1.24	7.97	3.24	3.75	1.24	0.069	0.877
1064	RI,TI	5-ethyldihydro-2(3h)-furanone	4.89 a	1.80 b	0.00 b	2.76	1.87	0.53	0.000	0.116
1099	TI	dipropenyldisulfide	2.28	6.33	1.93	2.92	4.23	1.01	0.152	0.402
		Microorganisms Esterification (0.3%)								
842	RI,TI	butanoic acid 1-methylethyl ester	4.77 a	5.33 a	0.00 b	3.37	3.62	0.83	0.017	0.851
1034	TI	hexanoic acid 1-methylethylester	5.06 a	0.5 b	2.49 ab	1.96	3.46	0.71	0.032	0.367
		Unknown Origin (5.4%)								
615	TI	acetic acid	121.43	85.20	110.91	108.35	102.96	18.09	0.693	0.870
1039	RI,TI	1-limonene	2.37	0.00	0.00	0.37	1.27	0.64	0.285	0.600

[a] RI = retention index. [b] Method of identification: RF, mass spectrum and retention time identical with a reference compound; RI, mass spectrum and retention index from literature in accordance; TI, tentative identification by mass spectrum. [c] GEN1 = IB × DU1; GEN2 = DU1 × IB; GEN3 = DU2 × IB. Different letters in the same row (a, b) indicate significant statistical differences (Tukey's Test, $p < 0.05$). [d] SEM = standard error of the mean.

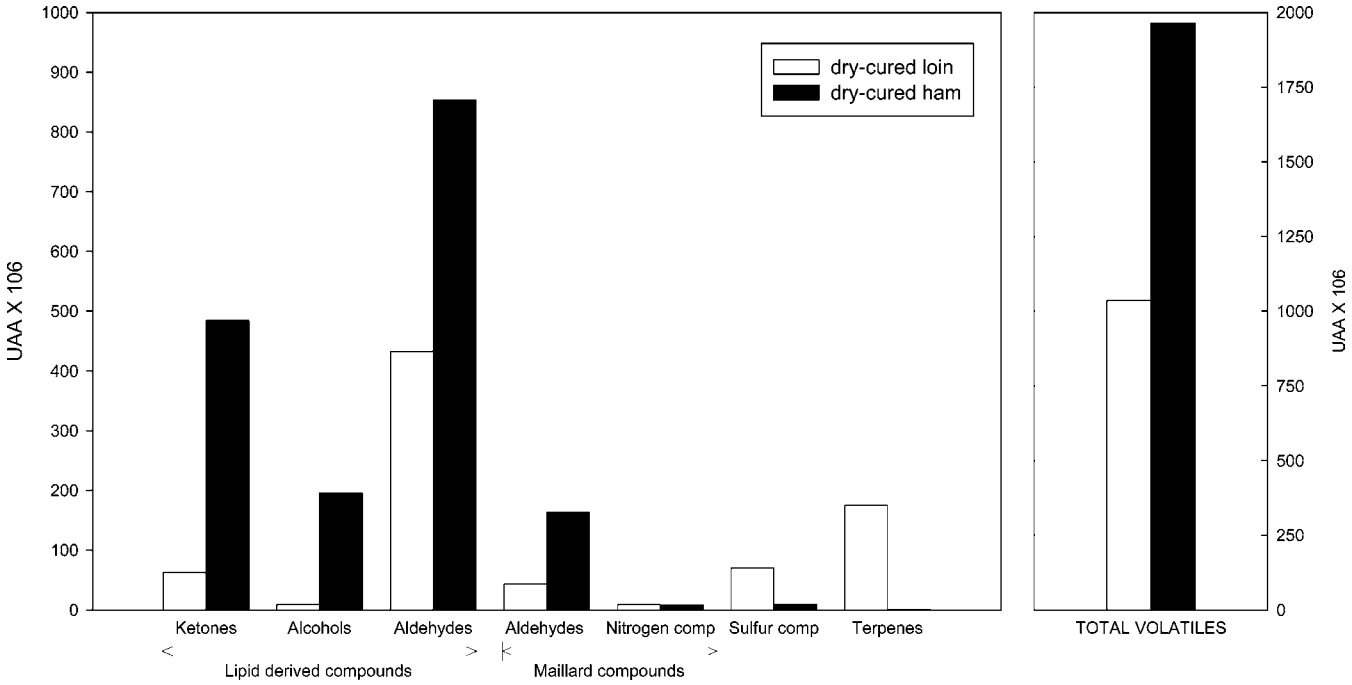

Figure 1. Volatile compounds (total volatile compounds and classified according to their origin and chemical nature) detected in dry-cured loin and ham.

protein derived compounds and the reaction between them to form Maillard volatile compounds would be enhanced in dry-cured ham. On the other hand, dry-cured loin had a higher content of certain volatile compounds from the pickling mixture, such as sulfur compounds and terpenes, which show strong aromatic notes and could play an important role in the overall aroma notes of this meat product.

Volatile compounds were classified according to their most likely origin in spite of the difficulties to establish the origin of some compounds. Volatile compounds were divided in lipid derived volatile compounds, which comprise acids, ketones, alcohols, aldehydes, and hydrocarbons, whereas volatile compounds formed via the Maillard reaction include heterocyclic nitrogen and sulfur compounds and non-heterocyclic compounds, such as Strecker aldehydes and hydroxyketones, as well as aliphatic disulfides. Besides, in dry-cured loin, volatile compounds from spices were detected, mainly terpenes and some sulfur compounds.

Some of these compounds detected have been previously described by Carrapiso et al. (*8*) as odor-active compounds in Iberian dry-cured ham such as 2-methylpropanal, 3-methylbutanal, 2-methylbutanal, 2-pentanone, pentanal, hexanal, 2-heptanone, heptanal, 2-heptenal, oct-1-en-3-ol, and octanal.

Lipid derived compounds were the main group of compounds in dry-cured loins and hams. In dry-cured loins, lipid derived compounds accounted for more than 55% of the total volatiles, and they accounted for more than 80% in dry-cured hams.

In general, acids are generated by lipid oxidation reactions. The origin of acetic acid, which was the most abundant acid compound detected in dry-cured ham, is not clear. Some authors have reported that it is originated by the fermentation of sugars by microorganisms (*15*) and others by the Maillard reaction (*16*). However, the importance of acetic acid in dry-cured ham may be limited, because this compound was not described as an odor-active compound of Iberian ham (*8*). In dry-cured loin, the importance of acetic acid could be higher than in dry-cured ham, since it could participate in its spiced characteristic aroma. Mateo et al. (*17*) have detected a wide variety of acids in Spanish paprika (an ingredient of the rubbing mixture), and especially acetic acid, which was also one the most abundant compounds detected in dry-cured loin.

The most abundant ketone detected in both products was propan-2-one. 2- Ketones have been abundantly isolated in dry-cured products, including dry-cured loin (*4*) and dry-cured ham (*9*). They have also been associated with the aroma of molded-surface cheeses (*18*), so their contribution to the overall cured flavor could be important. 2-Ketones may arise from fatty acids by chemical (autooxidation) or enzymatic (β-oxidation) oxidation of free-fatty acids by molds.

Aliphatic linear alcohols generally result from the degradation of lipid hydroperoxides (*19*). Dry-cured ham showed a wide variety of these compounds, detected in higher number and content than in dry-cured loin. Muriel et al. (*4*) reported that alcohols were the most important chemical group in dry-cured loin, in contrast to the present study in which two alcohols from lipid oxidation were detected.

Straight-chain aliphatic aldehydes are typical products of lipid oxidation. They have low odor threshold values and play an important role in the flavor of dry-cured ham and loin (*4*, *11*, *20*). Hexanal was the most abundant compound detected in both products. It is considered the main volatile derived from oxidation of *n*-6 fatty acids such as linoleic and arachidonic acids. The large amount of hexanal found in matured hams is considered a distinctive trait of these products, but in a high extent, this compound has been related to the development of rancid flavors in Iberian ham (*11*). The level of hexanal in dry-cured ham was approximately twice higher than in dry-cured loins (673×10^6 UA vs 380×10^6 UA), which is in accordance with the longer ripening time and the development in a higher extent of the lipid oxidation reactions in the former. Conversely, heptanal and octanal, which arise from oleic acid, add pleasant notes to the aroma of dry-cured products (*8*).

Aliphatic and aromatic hydrocarbons with origin in lipid oxidation reactions (*21*) were isolated in both dry-cured products. The contribution of these compounds to the aroma of dry-cured products is different. Aliphatic hydrocarbons, because of their high threshold value, are not important contributors to the aroma of these products, while aromatic hydrocarbons,

because of their abundance, could play an important role in the aroma of dry-cured loin and ham (*22*). Several authors have reported the presence of aromatic hydrocarbon compounds in dry-cured loin (*4*) and ham (*23*). They may play an important role in the overall flavor of meat products because some of them, such as ethylbenzene and methylbenzene, have been reported to be important to distinguish volatile profiles from different types of dry-cured hams (*23*).

Volatiles arising from the Maillard reaction accounted for ~18% (dry-cured loin) and ~13% (dry-cured ham) of the total volatiles. Most of them were branched short-chain aldehydes and their corresponding alcohols. The origin of branched aldehydes in dry-cured products is in Strecker degradation reactions of amino acids (*12*), which is an important pathway associated to the Maillard reaction. In the Strecker reaction, amino acids are decarboxylated and deaminated, forming aldehydes, while dicarbonyls formed in the Maillard reaction are converted to amino ketones or amino alcohols, which can also react with themselves or with other compounds, providing a wide variety of aromatic compounds (*13*).

Although many studies have been carried out about Iberian products' flavor, volatile compounds with origin in the Maillard reaction have never been quantified. The high proportion of volatiles from the Maillard reaction could be related with the high acceptability of these products, since Maillard compounds, which had very low threshold values, add pleasant aroma notes (*13*). Previous authors (*10*) have reported the importance of these compounds and a reduction of the lipid oxidation compounds in dry-cured hams with ripening time, because of the reaction of lipid oxidation products with other compounds, increasing the complexity of the volatile profile. This could be related with the intricacy of overall aroma and with the different aroma notes of the flavor of these products (*8*). On the other hand, the higher amounts and numbers of compounds with origin in protein degradation in dry-cured ham with respect to the loin show the implication of Maillard compounds in the aroma formation of the latter with respect to the former.

2- and 3-Methylbutanal are products of the Strecker degradation of the amino acids isoleucine and leucine, while benzeacetaldehyde comes from the amino acid phenylalanine (*24*) and acetaldehyde comes from the amino acid cysteine (*13*). 2- and 3-Methylbutanal, which were abundantly isolated in dry-cured ham, are linked to long ripening processes and, because of their low threshold values (*24*) and pleasant "cured" flavors, contribute positively to the dry-cured ham flavor (*10*).

Some branched-chain acids detected in both meat products, such as 2-ethylpropanoic acid, 3-methylbutanoic acid, and 2-methylbutanoic acid, have been identified as products of the microbial metabolism of valine, leucine, and isoleucine, respectively (*25*). Some authors have attributed the origin of these compounds to the action of molds, such as in dry-fermented meat products (*26*). The contribution of molds to the flavor of dry-cured ham has been previously reported (*27*), since different molds strive on the surface of dry-cured loin and ham. Proteolysis and lipolysis by the endogenous and microbial enzymes seem to play a decisive role in the generation of flavor precursors in dry-cured meat products (*27*). However, these compounds could also be originated by oxidation from their respective Strecker aldehydes (i.e., 2-methylbutanal would come from the degradation of the amino acid isoleucine, and by oxidation, 2-methylbutanoic acid would be formed). Ventanas et al. (*28*) reported that, under ripening conditions of dry-cured products, the formation of Strecker compounds is possible without the participation of microorganisms.

Alcohols such as propan-2-ol have their origin in amino acids by means of Strecker degradation reactions (*29*). 3-Methyl-1-butanol seems to play an important role in the overall and characteristic flavor of dry-cured hams (*23*). Its origin is controversial; some authors (*30*) have reported that this compound comes from 3-methylbutanal, a Strecker aldehyde, or from the microbial metabolism of leucine. However, Sánchez Peña et al. (*23*) postulated that its origin was from lipid oxidation, since they detected high levels of 3-methyl-1-butanol in muscle and also in subcutaneous fat where proteins are not abundant. Alcohols and aldehydes derived from Maillard reactions are relatively abundant and characteristic compounds of Iberian hams (*23*). In this sense, some of the most remarkable volatiles to the characterization of Iberian hams against hams from other breeds were 3-methyl-1-butanol and 3-methylbutanal (*23*), which are Strecker aldehydes.

Nitrogen and sulfur compounds have great importance in the overall flavor of meat products because of their very low threshold values (*24*). Except those from spices, nitrogen compounds come from the breakdown of proteins, free amino acids, and nucleic acids, while sulfur containing volatile compounds are derived from sulfur containing amino acids. Some nitrogen compounds, like pyrazines, have been abundantly isolated from dry-cured Iberian products (*4*, *8*). In addition, some furans were detected in dry-cured ham. Furans, because of their very low threshold value and their pleasant aroma (*24*), should contribute importantly to the desirable aroma of the dry-cured products.

Esters accounted for a small proportion of volatiles in dry-cured loin (0.8%) and dry-cured ham (0.3%). Most of them were ethyl esters, formed from ethanol and carboxylic acids by the action of microorganisms. Esters add fruity aroma notes (*31*). They have been isolated in Iberian dry-cured loin (*4*, *5*), in Iberian dry-cured ham (*9*, *10*), and in other dry-cured meat products such as in dry-fermented sausages (*26*, *31*−*33*).

In dry-cured loin, compounds derived from added spices lessen the volatiles derived from lipid oxidation, microbial metabolism, and Maillard reactions. Consequently, the flavor of the dry-cured loin is the result of a complex equilibrium between volatile compounds derived from both origins. In dry-cured loin, nearly 25% of volatiles had their origin in spices (Spanish paprika, oregano, and garlic), which were mainly terpenes and, to a lesser extent, aliphatic sulfur compounds. Three sulfur-derived compounds were detected, with dipropenyl disulfide (diallyl disulfide) being the most abundant. A wide variety of sulfur compounds derived from allicin are characteristic of garlic aroma (*34*), so it is likely that those had an important contribution to the overall aroma of dry-cured loin, since garlic is a potent aromatic ingredient. In addition, sulfur derivatives of propene have also been abundantly detected in dry-cured loin (*4*) and in other dry-fermented products manufactured with garlic (*32*, *33*).

Terpenes were abundant in dry-cured loin, while they were scarcely detected in dry-cured ham. They have well-defined odors in the literature, so alpha-pinene has been described to add a pine odor, while limonene and carene add lemon notes. In dry-cured ham, the presence of limonene has been associated with the pig diet (*9*, *23*). However, in dry-cured loin, the high content of terpenes suggests the origin in the spices added during the manufacture process (*4*). Taking into account the compounds of the seasoning mixture of dry-cured loin, terpenes have not been detected in dried Spanish paprika (*17*) nor in garlic (*32*), whereas they have been abundantly detected in oregano (*35*).

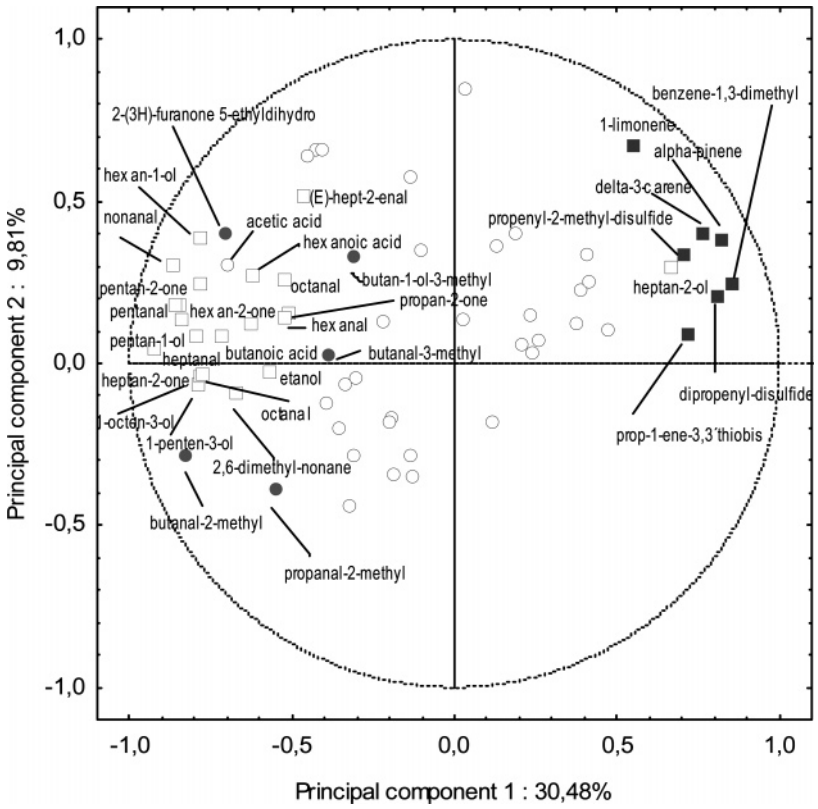

Figure 2. Loadings plot after principal component analysis of the volatile compounds from dry-cured loin and dry-cured ham in the plane defined by the two first principal components (PC1 and PC2): (○), volatile compounds not identified in the figure; (●), volatile compounds with origin in Maillard reaction; (■), volatile compounds with origin in species; and (□), volatile compounds with origin in lipid oxidation.

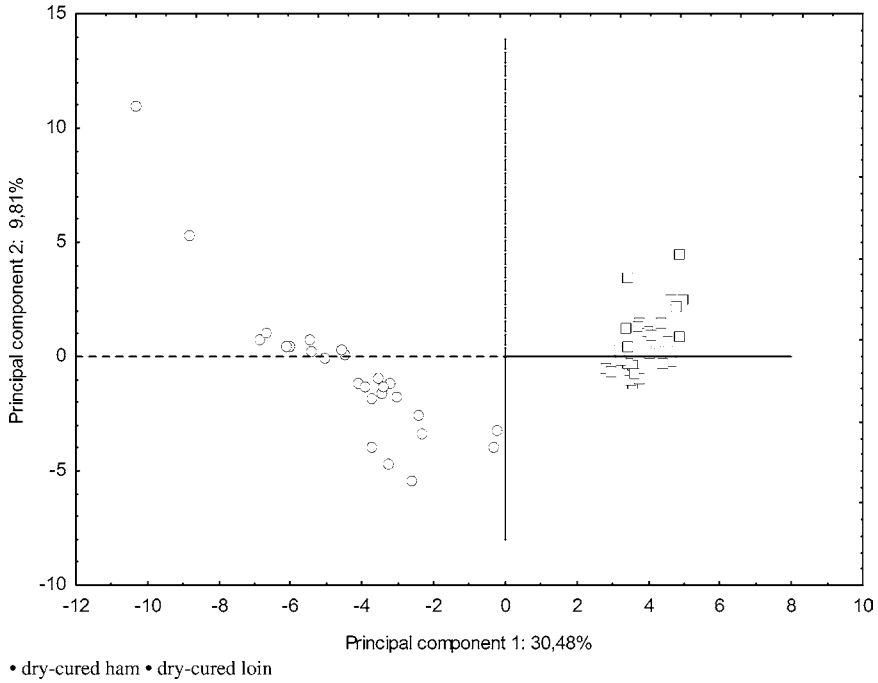

Figure 3. Scores plot after principal component analysis of the individuals in the plane defined by the two first principal components (PC1 and PC2): (○), dry-cured ham; (□), dry-cured loin.

Several pyrazines, methyl branched aldehydes, and their corresponding acids, with origin in the Maillard reaction, have been isolated from manufactured hot Spanish paprika by Mateo et al. (*17*). These compounds could also contribute, together with those formed from the raw material, to the pleasant flavor of Iberian dry-cured loin.

Volatile Profile of Iberian Dry-Cured Products as Affected by Pig Genotype. A few compounds showed differences between genotypes in both dry-cured products. In dry-cured loin, no differences were found in the amount of compounds derived from the pickling mixture, as all meat products followed the same manufacture process. Consequently, no significant dif-

ferences were found due to genotypes in the amount of terpenes and sulfur compounds. Some lipid-derived compounds such as ketones (octane-2,3-dione), aldehydes (hexanal, heptanal), and hydrocarbons (2-methylpentane) showed significant differences between genotypes. The significant ($p < 0.01$) differences in hexanal content between genotypes are outstanding, with them being higher in GEN3 than in GEN1 and GEN2. Hexanal has been considered a good indicator of the oxidative state of meat lipids (*24*) and flavor deterioration, since it is responsible for the rancid aroma in meat products when it is present at high concentrations (*11*). Thus, those authors found lower hexanal and TBA-RS numbers in Iberian dry-cured hams with less rancid flavors. However, heptanal, which adds pleasant flavors, was higher in GEN3 than in GEN1 and GEN2. The low number of compounds that showed differences between genotypes in dry-cured loin is in accordance with the results of Muriel et al. (*4*), who did not find differences in the volatile profiles of dry-cured loins from different Iberian genotypes. The compounds from the seasoning mixture in dry-cured loin have great importance on the overall flavor of this meat product, since sulfur and terpenes compounds were one-fourth of the aroma compounds detected. The spices added in the seasoning could have contributed to disguise the quality differences previously reported in the other parameters analyzed in fresh meat and in dry-cured loins (*36*, *37*), as the high level of the compounds from the seasoning may have reduced the differences in the volatile profile among genotypes.

In dry-cured ham, only eight compounds showed differences between batches. Hams from GEN1 showed significantly highest contents of 1-etoxi-heptan-2-one, hexan-1-ol, (*E*)-hepten-2-al, octanal, hexanoic acid-1-methylethyl ester, butanoic acid-1-methylethyl ester, and 5-ethyldihydro-2(3H)-furanone, while hams from GEN2 showed the significantly highest content of butanoic acid-1-methylethyl ester and hams from GEN3 showed the significantly highest content of 3-methyl-butanoic acid. The scarce differences between genotypes could be due to the similar fatty acid composition of intramuscular fat of *Biceps femoris* (*36*), because the changes in the lipids during processing are the main contributors to volatile flavor compounds formation (*8*, *11*).

Changes in fatty acid composition of IMF affect volatile compounds, especially aldehydes formed during the maturing of the piece (*7*, *11*). Cava et al. (*11*) reported that hams manufactured from raw meat with lower PUFA contents had lower hexanal content and were perceived as less rancid. Consequently, the differences found in hexanal content in dry-cured loin and the lack of differences in dry-cured ham would agree with the more marked differences of the fatty acids composition in the raw material for the manufacture of these meat products analyzed whenfresh (*36*).

Multivariate Analysis. A principal component analysis (PCA) was carried out to determine the relationship between the volatile compounds detected. PCA of these variables resulted in five significant factors that accounted for 56.9% of the variability. 30.48% and 9.81% of the variability is explained by principal components (PCs) #1 and #2, respectively.

Figure 2 shows the score plot of the different variables (coefficients of the eigenvectors) for the two first principal components (PC#1 and PC#2). Although all volatiles were included in the analysis, only volatile compounds that explained more variation of the data have been identified in the figure. In this plot, two groups of variables are clearly separated. In the positive axis on the PC#1, far from the origin and explaining an important part of the variation, are located volatile compounds isolated in dry-cured loin, mainly those compounds with origin in species, such as 1-limonene, alpha-pinene, delta-3-carene, dipropenyldisulfide, propenyl-2-methyldisulfide, while lipid derived compounds, such as aldehydes, ketones, and alcohols, and Maillard reactions compounds, such as branched aldehydes, alcohols, and furanones, were located in the negative axis on PC#1.

The distribution of the individuals on the two first PCs (**Figure 3**) shows two separate groups of points, corresponding to dry-cured loin and dry-cured ham, while no differences between genotypes were found (data not shown). Loins were located in the positive area of PC1, the same as terpenes and sulfur compounds in the variables' plot, so loins and terpenes and sulfur compounds are closely linked. On the other hand, hams were located in the negative area of PC1, so they were associated to lipid derived and Maillard compounds. Therefore, the volatile profile was the main factor to characterize adequately dry-cured loins and dry-cured hams because it did not differ between genotypes.

In conclusion, lipid derived volatiles and Maillard compounds were isolated in both meat products, although in dry-cured ham they were more abundant as a result of the longer ripening process and the greater complexity of the compounds formed. Additionally, volatiles with origin in the seasoning mixture were only isolated in dry-cured loin; therefore, the different manufacture process of these meat products characterizes their aromatic profile. However, pig genotype (reciprocal cross and Duroc sire line) slightly affected the volatile profile of Iberian × Duroc dry-cured meat products.

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