

Changes in SPME-extracted volatile compounds from Iberian ham during ripening

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RESUMEN

Cambios de compuestos volátiles extraídos por SPME en jamones Ibéricos durante la curación.

Un procedimiento de microextracción en fase sólida (SPME) en espacio de cabeza fue ensayado para estudiar la evolución de los compuestos volátiles durante la curación de Jamones Ibéricos de cerdos criados en régimen de montanera (en libertad, con bellotas y pasto disponible) y un sistema OH-Pienso (estabulados, con un pienso alto en ácido oléico). El efecto del tiempo de curación en los compuestos volátiles fue más marcado que el efecto de la alimentación. La mayoría de los compuestos volátiles afectados por el tiempo de curación fueron compuestos que proceden de la reacción de Strecker y Maillard, que se incrementaron significativamente ($p < 0.05$) durante el tiempo de curación. Esta tendencia fue clara para los compuestos volátiles: 2-metilpropanal, 2-metilbutanal, 2-metil-1-butanol, disulfuro de dimetilo, 2,6-dimetilpiracina + dihidro-2(3H)furanona y benzenometanol y, por tanto ellos podrían ser usados como indicadores del tiempo de maduración. Por el contrario, algunos compuestos derivados de la oxidación lipídica disminuyeron significativamente durante el procesado del jamón. El sistema de alimentación tuvo un ligero efecto en los compuestos volátiles, aunque fue significativo para algunos compuestos ($p < 0.05$). Algunos de ellos (ácido butanoico, 2,6-dimetilpiracina y 1-octen-3-ol) fueron más abundantes en jamones de Montanera que en jamones de Pienso. Estas pequeñas diferencias podrían ser importantes porque los compuestos involucrados han sido identificados como aromas del jamón Ibérico.

PALABRAS CLAVES: *Compuestos volátiles – Jamón Ibérico – Microextracción en fase sólida – Sistema de alimentación – SPME – Tiempo de curación.*

SUMMARY

Changes in SPME-extracted volatile compounds from Iberian ham during ripening.

A headspace SPME procedure was tested to study the evolution of volatile compounds during the ripening of Iberian hams from pigs reared in a Montanera system (outdoor-based, with acorn and pasture available) and a HO-Pienso system (indoor-based, with a high oleic acid concentrate). The effect of the ripening time on volatile compounds was more marked than the effect of feeding system. Most volatile compounds affected by the ripening time were compounds that come from Strecker and Maillard reactions, which

increased significantly ($p < 0.05$) during the ripening time. This trend was clear for the volatile compounds 2-methylpropanal, 2-methylbutanal, 2-methyl-1-butanol, disulfide dimethyl, 2,6-dimethylpyrazine + dihidro-2(3H) furanone and benzenemethanol and, therefore they could be used as indicators of the ripening time. Conversely, some of the lipid oxidation-derived compounds decreased significantly throughout ham processing. Feeding system had a weak effect on volatile compounds, but it was significant for some compounds ($p < 0.05$). Some of them (butanoic acid, 2,6-dimethylpirazine and 1-octen-3-ol) were more abundant in Montanera than in Pienso hams. These small differences could be important because they involve compounds that have been identified as Iberian ham odorants.

KEY-WORDS: *Feeding system – Iberian ham – Ripening time – Solid-phase microextraction – SPME – Volatile compounds.*

1. INTRODUCTION

Dry-cured Iberian ham is a product of great importance in Spain. Its typical flavour is an outstanding quality parameter and a major contributor to consumer acceptance (Ruiz *et al.*, 2002). Iberian ham sensory quality depends on raw meat characteristics, which are mainly influenced by genetic factors (Carrapiso *et al.*, 2003a; Carrapiso and García, 2008), rearing system (Cava *et al.* 2000a; Carrapiso *et al.*, 2003a), concentrate feeding composition (Ruiz *et al.*, 2005; Ventanas *et al.*, 2007; Jurado *et al.*, in press), montanera feeding period length (Carrapiso *et al.*, 2007), but also by processing conditions (Andrés *et al.*, 2004). Therefore, there are a lot of relevant factors that influence Iberian ham sensory characteristics, and that causes a great heterogeneity in the Iberian ham quality.

Currently the quality control is performed in raw meat but barely during Iberian ham process: the only compulsory control is a minimum ripening time (500 or 660 days according to the pieces weight) (RD 1469/2007). Ripening conditions and time are essential for flavour development, and in fact it has been demonstrated that ripening time affects flavour sensory traits: hams with longer ripening

times show higher odour and flavour features and lower rancidity values than hams with shorter ripening times (Ruiz *et al.*, 1999).

Volatile compounds are the main contributors to flavour of meat products (Mottram, 1998), and they are generated through the chemical reactions (lipid autooxidation, Strecker and Maillard reactions) which take place throughout Iberian ham ripening (García *et al.*, 1991). Therefore, volatile compounds could be suitable to estimate the flavor features, and therefore Iberian ham quality. By knowing the volatile compound profile, it would be possible to predict the current ripening time or if the pieces have been ripened for the minimum required ripening time. In addition, they could allow estimate the feeding system used to fatten the animals.

Solid phase microextraction (SPME) is an effective tool for analysing the volatile compounds. This technique is sensitive, selective, fast, low-cost, solvent free, easy to handle and also compatible with low detection limits. In addition, as a low extraction temperature can be used, SPME could give a better estimation of the aroma profile as it is perceived by the human nose (reviewed by Kataoka *et al.*, 2000; Brunton *et al.*, 2000). SPME has been widely used to extract volatile compounds in several foods and drinks (Chin *et al.*, 1996; Elmore *et al.*, 1997; Bazemore *et al.*, 1999; Marsili, 1999) and also in dry-cured ham (Gianelli *et al.*, 2002; García-Esteban *et al.*, 2004) and Iberian ham (Ruiz *et al.*, 1998, 2001; Andres *et al.*, 2002; Ramirez and Cava, 2007).

Andrés *et al.* (2002, 2007) investigated the changes in the volatile compounds of Iberian ham during ripening by using a direct-extraction device for the SPME extraction, showing large fluctuations in the volatile compound abundances. Although the procedure has the unquestionable advantage of being minimally destructive, it does not provide an accurate control of the temperature during the volatile compound extraction. The SPME-extraction is greatly influenced by temperature (Prosen and Zupancic-Krajc 1999; Kataoka *et al.*, 2000). The aim of this study was to research the changes during ripening in the volatile compounds extracted by using a conventional SPME procedure that provides an accurate control of the extraction conditions, and to propose volatile compounds that could be used as indicators of the flavour development throughout processing or the feeding system.

2. MATERIALS AND METHODS

2.1. Raw Iberian hams

Forty legs were obtained from Iberian pigs reared in confinement on a high oleic acid concentrate (Pienso hams) (for details on composition, see Muriel *et al.*, 2002). Other forty legs were obtained from Iberian pigs reared in a free-range system during the fattening period (75 days before slaughter), the only fed source being acorn and pasture (Montanera hams). All the animals were slaughtered at about 145 ± 10 Kg (mean ± standard deviation).

2.2. Processing of the dry-cured Iberian hams

The hams were processed in controlled humidity and temperature rooms, first in a local factory (the first 100 days) and then in the Department of Food Science (University of Extremadura) by applying the usual temperature and relative humidity values of the tradicional processing (for details, see Jurado *et al.*, 2007). Conditions applied during Iberian ham ripening are shown in Table 1. The whole process lasted 722 days.

2.3. Sampling

The hams were sampled at five different moments of processing: at day 120 (end of postsalting), day 230 (end of the drying stage), day 361, day 516, and day 722 (end of the first third, the second third and the end of the cellar phase). Five Montanera hams and five Pienso hams were sampled each time, except at the end of processing, when twenty Montanera hams and twenty Pienso hams were sampled.

Samples were obtained by extracting a cilinder (sized 10 × 2.5 cm) using a stainless steel tube with a cutting edge and they mainly included the *Semimembranosus* and *Biceps femoris* muscles. Samples were vacuum-packaged and frozen at -80 °C until being analysed. Subcutaneous and intermuscular fat were removed just before the analyses.

2.4. Volatile compound extraction procedure

A SPME fibre coated with Carboxen-poly(dimethylsiloxane) (CAR/PDMS) (75µm thickness,

Table 1
Conditions applied during Iberian ham ripening and the five different times of ham sampling

| | Temperature | Relative humidity | Days | Sampling |
|---------------|------------------|-------------------|--------------------|-----------------------|
| Salting | 2-3 °C | 95% | 0-10/11 (a day/kg) | |
| Postsalting 1 | 2-3 °C | 85% | 11-63 | |
| Postalting 2 | 10 °C | 75% | 63-120 | Day 120 |
| Drying | from 10 to 30 °C | from 75% to 65% | 121-230 | Day 230 |
| Cellar | 19 °C | 70% | 231-722 | Days 361, 516 and 722 |

Supelco Co., Bellefonte, U.S.A.) was used to extract the volatiles. This type of fibre was chosen because of its high sensitivity (Brunton *et al.*, 2000) and it has been recommended to extract the dry-cured ham volatile compounds (Gianelli *et al.*, 2002; García-Esteban *et al.*, 2004). Prior to extraction analysis, the SPME fibre was preconditioned at 280 °C for 45 min in the GC injection port.

Samples were ground with a commercial grinder. 1 g was weighted into a 4 ml vial, which was screw-capped with a laminated Teflon-rubber disk. The fibre was inserted into the sample vial through the septum and exposed to headspace for 30 min. The vial was kept in a water bath at 35 °C during the extraction procedure.

2.5. Gas chromatography-mass spectrometry

The SPME fibre was inserted (and maintained during the whole chromatographic run) in the injection port in the splitless mode (at 280 °C) of a HP 6890GC series II gas chromatograph (Hewlett-Packard, U.S.A.) coupled to a mass-selective detector (HP-5973A, Hewlett-Packard, U.S.A.). Volatiles were separated using a HP-5 bonded-phase fused-silica capillary column (5% phenyl-methylsilicone, 50 m × 0.32 mm I.D., film thickness 1.05 µm, Hewlett-Packard, U.S.A.). The oven temperature program was isothermal for 10 min at 40 °C, raised to 250 °C at the rate of 5 °C min⁻¹, 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, with a multiplier voltage of 1756V, collecting data at a rate of 1 scan s⁻¹ over the m/z range of 30 to 500. *n*-Alkanes (Sigma, Saint Louis MO, USA) were run under the same chromatographic conditions to calculate the Kovats indexes.

Compounds were tentatively identified by comparing mass spectra with those contained in the NIST/EPA/NIH and Wiley libraries and by comparison of the calculated Kovats indexes with those reported in the literature (Kondjoyan and Berdagué, 1996; Elmore *et al.*, 2000; Carrapiso *et al.*, 2003b). Results were expressed as area units (AU).

2.6. Data analysis

The effect of processing time (120, 230, 361, 516 and 722 days), rearing conditions (Montanera vs Pienso) and the interaction were analysed by using a two-way analysis of variance with interaction by the GLM procedure. When significant differences among the means of each group appeared, they were checked by using the Tukey test. A factor analysis (using principal components analysis as the method for factor extraction) was applied to evaluate the relations between variables (Hair *et al.*, 1998). Statistic analyses were performed by means of the SPSS version 15.0.

3. RESULTS AND DISCUSSION

Among the identified compounds (n = 70), only those appearing in all the samples of at least one ripening stage were selected to be researched during the ripening. The 36 volatile compounds selected are included in Table 2. Some of them appeared in coelution with another compound: pentanal and heptane, hexanal and octane, 2,6-dimethylpyrazine and dihydro 2(3H)-furanone.

All these volatile compounds have been previously identified in fully ripened Iberian ham (García *et al.*, 1991), and previous researches on this product have investigated the effect on them of the rearing system (López *et al.*, 1992; Carrapiso *et al.*, 2002a; Carrapiso *et al.*, 2003b), ripening time (Ruiz *et al.*, 1999; Andrés *et al.*, 2002), the bone taint spoilage of hams (García *et al.*, 2000). Some

Table 2
Selected volatile compounds

| Volatile Compounds | LRI ^a | Origin ^b |
|--------------------------------|------------------|---------------------|
| Acetaldehyde | <500 | aa |
| Metanethiol | <500 | aa |
| 2-Propanone | <500 | lipids |
| 2-Methylpropanal | 547 | aa |
| Acetic acid | 597 | aa ^c |
| 3-Methylbutanal | 651 | aa |
| 2-Methylbutanal | 662 | aa |
| 2-Pentanone | 688 | lipids |
| Pentanal | 697 | lipids |
| Heptane | 700 | lipids |
| 3-Methyl-1-butanol | 738 | aa |
| 2-Methyl-1-butanol | 743 | aa |
| Dimethyl disulfide | 752 | aa |
| 1-Pentanol | 770 | lipids |
| Butanoic acid | 781 | aa ^c |
| 2,3- Butanodiol | 789 | lipids |
| Octane | 800 | lipids |
| Hexanal | 801 | lipids |
| 3-Methylbutanoic acid | 837 | aa |
| 1-Hexanol | 869 | lipids |
| 2-Heptanone | 888 | lipids |
| Heptanal | 900 | lipids |
| 3-Methylthiopropanal | 905 | aa |
| 2,6-Dimethylpyrazine | 913 | aa |
| Dihydro 2(3H)-furanone | 914 | aa ^c |
| Benzaldehyde | 968 | aa |
| 1-Octen-3-ol | 979 | lipids |
| 2-Pentylfuran | 991 | lipids |
| Octanal | 1003 | lipids |
| Benzenemethanol | 1039 | aa |
| Benzeneacetaldehyde | 1052 | aa |
| 5-Ethylidihydro-2(3H)-furanone | 1060 | aa ^c |
| 1-Octanol | 1068 | lipids |
| Nonanal | 1104 | lipids |
| Benzeneethanol | 1123 | aa |
| 5-Butylidihydro-2(3H)-furanone | 1291 | aa ^c |

^a LRI values: linear retention indices on HP-5 capillary column.

^b Origin of volatile compounds (Belitz y Grosh, 1992; Mottram, 1998; Pripis-Nicaulau *et al.*, 2000): **aa**: volatile compounds coming from amino acids generated through Strecker and Maillard reactions. **Lipids**: volatile compounds coming from lipid oxidation.

^c Also generated through lipid oxidation reactions.

of these compounds contribute to the overall ham flavour as they have been reported as odorant of Iberian hams (Carrapiso *et al.*, 2002b; Carrapiso and García, 2004), and also of other dry-cured hams (Flores *et al.*, 1997; Blank *et al.*, 2001) and meat products (Mottram, 1998; Meynier *et al.*, 1999; Timón *et al.*, 2004).

Among the 36 compounds included in Table 2. 16 of them have been associated with lipid oxidation reactions, and even some of them are lipid oxidation markers (Shahidi *et al.*, 1987; Cava *et al.*, 1999; Shahidi, 2001; O'Sullivan *et al.*, 2003); 15 compounds are generated from amino acids through Strecker and Maillard reactions; and 5 of them could generate through lipid oxidation or Strecker and Maillard reactions (Belitz and Grosh, 1992; Mottram, 1998; Pripis-Nicaulau *et al.*, 2000) (Table 2).

3.1. Changes throughout Iberian ham processing

Volatile compound profiles were markedly different throughout processing. At the end of postsalting, (day 120) the most abundant volatile compounds were the coelution of hexanal and octane, 2-propanone, acetic acid, 1-pentanol, nonanal and the coelution of pentanal and heptane. However, in the finished product 3-methylbutanal was the most abundant volatile compound, followed by hexanal and octane, acetic acid, 2-methylbutanal, 2-propanone and 2-methylpropanal. In summary, in the first sampling time (day 120), the compounds from lipid oxidation dominated the volatile compound profile, whereas in the finished product volatile compounds from Strecker reactions (3-methylbutanal, 2-methylbutanal, 2-methylpropanal) became the most abundant volatile compounds.

With regard to the quantitative changes in the volatile compounds throughout processing, the SPME-GC-MS procedure allowed to find a significant effect of the ripening time on the total chromatographic area counts. A steady increase of the total area was found from day 120 until day 516, and then a slight but not significant decrease appeared (Table 3). This trend agrees with a previous study focused on the evolution of Iberian ham volatile compounds extracted by using a direct-extraction-SPME method that did not allow an accurate control of temperature extraction (Andrés *et al.* 2002).

Most volatile compounds (24 of 36) were significantly influenced by the ripening time (Table 3). Most of them are compounds that come from Strecker and Maillard reactions, and only six compounds are generated from lipid oxidation reactions (1-pentanol, 1-hexanol, 2-heptanone, octanal, 1-octanol) (Table 2).

With regard to the Strecker and Maillard volatile compounds, they increased significantly during the ripening time (Table 3). There was a marked increase from day 120 to day 230 in these compounds and, in fact, some of these compounds were not detected

until day 230 (methanethiol, 2-methylpropanal, dimethyl disulfide, 3-methylthiopropional, benzenemethanol, benzeneacetaldehyde, 5-ethylidihydro-2(3H)-furanone), and even some of them were not detected until day 361 (benzeneethanol and 5-butyldihydro-2(3H)-furanone) (Table 3). The volatile compounds also increased after day 230, and some of them (2-methylpropanal, 2-methylbutanal, 2-methyl-1-butanol, disulfide dimethyl, 2,6-dimethylpyrazine + dihidro-2(3H)furanone and benzenemethanol) reached the largest levels at the last stage of processing (Table 3), and therefore they could be used as indicators of the ripening time. By studying these compounds it would be possible to estimate the Iberian ham current ripening time or if the pieces have been ripened for the minimum ripening time established for Iberian hams (RD 1469/2007). It is interesting to take into account the evolution of 2-methylbutanal which should reach values above 1400 UAA in the last stage of processing, because it would indicate a processing longer than 660 days.

On the other hand, these results would reveal a steady development of chemical reactions involved in the generation of Strecker and Maillard derived volatile compounds, including protein degradation, throughout the whole ripening stage, resulting in an increase in these compounds. In a previous study, we demonstrated that an increase of free amino acids was followed by the generation of amino acids derived volatile compounds (Jurado *et al.*, 2007).

This trend is in agreement with results by Ruiz *et al.*, (1999), who reported that branched aldehydes (2 and 3-methylbutanal) were more abundant in the more aged Iberian hams. Hinrichsen and Pedersen (1995) have also found that Parma hams with a ripening time of 125 days showed lower levels of 3-methylbutanal, 2-methylbutanal, 2-methylpropanal than those with a ripening time of 485 days. However, Andres *et al.* (2002), who monitored some of the branched aldehydes (2 and 3-methylbutanal) throughout Iberian ham processing by using a direct-extraction-SPME method, did not find a clear pattern in the evolution of these compounds. In the cited study, the extraction of volatile compounds was performed at the temperature of the chamber where hams were kept in each processing time, which probably led to obtain more variability in the volatile compounds extraction than in the present study. In fact, for accurate and precise analysis, a control of the extraction temperature is essential (reviewed by Kataoka *et al.*, 2000), and also it has been previously reported when sampling dry cured Iberian ham (Ruiz *et al.*, 1998).

Conversely to the Strecker and Maillard volatile compounds, five compounds derived from lipid oxidation reactions (1-pentanol, 1-hexanol, octanal, 1-octen-3-ol, 1-octanol) decreased significantly during ripening, reaching the lowest levels in the last stage of processing. Only 2-heptanone, also a compound from lipid oxidation, increased during the ripening time (Table 2).

Table 3
Abundance (area units $\times 10^{-5}$) of volatile compounds (means \pm standard deviations) obtained by using a SPME method during the ripening of Iberian hams obtained in Montanera (outdoors) and in Pienso (indoors) systems, and results from a two-way ANOVA with interaction^A

| | Day 120 | | Day 230 | | Day 361 | | Day 516 | | Day 722 | | p | | |
|--------------------------------|------------------------------|-----------------------------|-------------------------------|-------------------------------|--------------------------------|--------------------------------|-------------------------------|-------------------------------|--------------------------------|--------------------------------|--------|--------|-------|
| | Montanera | Pienso | Montanera | Pienso | Montanera | Pienso | Montanera | Pienso | Montanera | Pienso | T | A | A*T |
| Acetaldehyde | 17 \pm 19 ^b | 26 \pm 27 ^b | 30 \pm 15 ^b | 49 \pm 36 ^b | 53 \pm 15 ^b | 70 \pm 45 ^b | 118 \pm 77 ^{ab} | 235 \pm 91 ^a | 92 \pm 60 ^b | 97 \pm 71 ^b | <0.001 | 0.044 | 0.160 |
| Metanethiol | nd ^b | nd ^b | 7 \pm 6 ^{ab} | 8 \pm 3 ^{ab} | 24 \pm 12 ^{ab} | 15 \pm 4 ^{ab} | 30 \pm 5 ^a | 26 \pm 26 ^{ab} | 26 \pm 13 ^{ab} | 30 \pm 18 ^a | <0.001 | 0.683 | 0.744 |
| 2-Propanone | 1519 \pm 873 | 2114 \pm 664 | 1765 \pm 671 | 1216 \pm 248 | 1365 \pm 277 | 1164 \pm 473 | 1888 \pm 736 | 1082 \pm 742 | 1558 \pm 776 | 1440 \pm 488 | 0.439 | 0.218 | 0.172 |
| 2-Methylpropanal | nd ^c | nd ^c | 294 \pm 409 ^{abc} | 107 \pm 59 ^{bc} | 320 \pm 88 ^{abc} | 354 \pm 235 ^{abc} | 758 \pm 396 ^{abc} | 421 \pm 222 ^{abc} | 1052 \pm 482 ^a | 797 \pm 459 ^{ab} | <0.001 | 0.158 | 0.718 |
| Acetic acid | 685 \pm 182 | 836 \pm 340 | 891 \pm 49 | 829 \pm 467 | 1007 \pm 363 | 902 \pm 171 | 1564 \pm 628 | 1205 \pm 173 | 2678 \pm 1821 | 2206 \pm 1608 | <0.001 | 0.639 | 0.964 |
| 3-Methylbutanal | 104 \pm 27 ^c | 122 \pm 93 ^c | 2013 \pm 2459 ^{bc} | 3179 \pm 3334 ^{bc} | 5073 \pm 3511 ^{abc} | 6350 \pm 2631 ^{abc} | 10013 \pm 5352 ^a | 6835 \pm 2196 ^{ab} | 5496 \pm 2438 ^{abc} | 6005 \pm 4398 ^{abc} | <0.001 | 0.964 | 0.590 |
| 2-Methylbutanal | 102 \pm 47 ^d | 100 \pm 50 ^d | 519 \pm 448 ^{abcd} | 368 \pm 71 ^{cd} | 708 \pm 121 ^{abcd} | 848 \pm 610 ^{abcd} | 1171 \pm 376 ^{abc} | 934 \pm 344 ^{abcd} | 1682 \pm 647 ^a | 1462 \pm 576 ^{ab} | <0.001 | 0.499 | 0.863 |
| 2-Pentanone | 43 \pm 7 | 67 \pm 50 | 155 \pm 155 | 388 \pm 470 | 338 \pm 296 | 196 \pm 91 | 618 \pm 1125 | 117 \pm 196 | 137 \pm 106 | 110 \pm 138 | 0.158 | 0.359 | 0.214 |
| Pentanal + Heptane | 302 \pm 160 | 337 \pm 168 | 798 \pm 859 | 436 \pm 185 | 726 \pm 595 | 839 \pm 619 | 585 \pm 210 | 539 \pm 141 | 754 \pm 405 | 577 \pm 334 | 0.093 | 0.419 | 0.687 |
| 3-Methyl-1-butanol | 117 \pm 42 ^c | 70 \pm 59 ^c | 176 \pm 124 ^{bc} | 88 \pm 37 ^c | 225 \pm 70 ^{bc} | 361 \pm 363 ^{abc} | 924 \pm 455 ^a | 533 \pm 105 ^{abc} | 610 \pm 217 ^{abc} | 715 \pm 424 ^{ab} | <0.001 | 0.471 | 0.200 |
| 2-Methyl-1-butanol | 9 \pm 10 ^b | 9 \pm 14 ^b | 36 \pm 28 ^{ab} | 15 \pm 8 ^b | 48 \pm 14 ^{ab} | 93 \pm 85 ^{ab} | 133 \pm 23 ^{ab} | 109 \pm 26 ^{ab} | 229 \pm 116 ^a | 230 \pm 142 ^a | <0.001 | 0.991 | 0.949 |
| Dimethyl disulfide | nd | nd | 36 \pm 50 | 25 \pm 24 | 153 \pm 138 | 65 \pm 36 | 137 \pm 47 | 122 \pm 121 | 287 \pm 270 | 251 \pm 195 | <0.001 | 0.556 | 0.986 |
| 1-Pentanol | 511 \pm 307 ^{abc} | 646 \pm 322 ^{ab} | 847 \pm 861 ^a | 369 \pm 196 ^{bc} | 204 \pm 56 ^c | 182 \pm 33 ^c | 131 \pm 31 ^c | 133 \pm 61 ^c | 106 \pm 47 ^c | 110 \pm 106 ^c | <0.001 | 0.167 | 0.093 |
| Butanoic acid | 279 \pm 76 | 282 \pm 125 | 329 \pm 104 | 232 \pm 85 | 334 \pm 40 | 279 \pm 97 | 381 \pm 49 | 361 \pm 49 | 455 \pm 186 | 262 \pm 88 | 0.200 | 0.035 | 0.994 |
| 2,3-Butanediol | nd | 20 \pm 27 | 121 \pm 210 | nd | 19 \pm 42 | 69 \pm 79 | 77 \pm 101 | 47 \pm 58 | 231 \pm 311 | 200 \pm 577 | 0.321 | 0.816 | 0.989 |
| Hexanal + Octane | 5630 \pm 4010 | 6161 \pm 3989 | 3618 \pm 2393 | 3182 \pm 1758 | 3801 \pm 4244 | 4060 \pm 2260 | 2894 \pm 1047 | 2979 \pm 907 | 2963 \pm 1501 | 3746 \pm 2945 | 0.082 | 0.735 | 0.979 |
| 3-Methylbutanoic acid | 25 \pm 16 | 21 \pm 16 | 41 \pm 23 | 50 \pm 33 | 85 \pm 60 | 80 \pm 49 | 165 \pm 122 | 66 \pm 25 | 195 \pm 217 | 91 \pm 57 | 0.054 | 0.247 | 0.574 |
| 1-Hexanol | 207 \pm 57 ^b | 199 \pm 155 ^b | 532 \pm 627 ^a | 212 \pm 98 ^b | 157 \pm 92 ^b | 208 \pm 101 ^b | 131 \pm 43 ^b | 120 \pm 31 ^b | 125 \pm 56 ^b | 95 \pm 54 ^b | <0.001 | 0.081 | 0.049 |
| Heptanone | 13 \pm 6 | 11 \pm 3 | 34 \pm 15 | 43 \pm 22 | 44 \pm 23 | 47 \pm 33 | 50 \pm 44 | 41 \pm 26 | 87 \pm 64 | 86 \pm 102 | 0.014 | 0.992 | 0.999 |
| Heptanal | 165 \pm 104 | 215 \pm 124 | 211 \pm 103 | 220 \pm 16 | 229 \pm 152 | 263 \pm 143 | 207 \pm 58 | 182 \pm 30 | 173 \pm 84 | 206 \pm 122 | 0.661 | 0.500 | 0.947 |
| 3-Methylthiopropenal | nd ^b | nd ^b | nd ^b | 5 \pm 11 ^b | 10 \pm 3 ^b | 10 \pm 8 ^b | 50 \pm 29 ^a | 33 \pm 9 ^{ab} | 33 \pm 24 ^{ab} | 28 \pm 23 ^{ab} | <0.001 | 0.522 | 0.797 |
| 2,6-Dimethylpyrazine | 29 \pm 2 ^d | 17 \pm 5 ^d | 40 \pm 17 ^{cd} | 34 \pm 9 ^{cd} | 80 \pm 3 ^{bcd} | 90 \pm 82 ^{bcd} | 139 \pm 32 ^{ab} | 70 \pm 25 ^{bcd} | 176 \pm 49 ^a | 109 \pm 31 ^{abc} | <0.001 | 0.008 | 0.017 |
| + Dihydro 2(3H)-furanone | | | | | | | | | | | | | |
| Benzaldehyde | 15 \pm 20 ^c | 35 \pm 37 ^{bc} | 156 \pm 171 ^{abc} | 85 \pm 44 ^{bc} | 163 \pm 87 ^{abc} | 160 \pm 119 ^{abc} | 371 \pm 148 ^a | 246 \pm 141 ^{ab} | 211 \pm 117 ^{abc} | 185 \pm 124 ^{abc} | <0.001 | 0.196 | 0.662 |
| 1-Octen-3-ol | 109 \pm 66 ^a | 90 \pm 26 ^a | 110 \pm 110 ^a | 56 \pm 28 ^{ab} | 56 \pm 35 ^{ab} | 55 \pm 19 ^{ab} | 56 \pm 18 ^{ab} | 39 \pm 10 ^b | 58 \pm 21 ^{ab} | 49 \pm 21 ^{ab} | 0.001 | 0.031 | 0.508 |
| 2-Pentylfuran | 4 \pm 3 | 5 \pm 4 | 10 \pm 8 | 5 \pm 8 | 4 \pm 1 | 36 \pm 72 | 5 \pm 1 | 4 \pm 4 | 28 \pm 58 | 8 \pm 7 | 0.686 | 0.899 | 0.368 |
| Octanal | 157 \pm 71 | 178 \pm 67 | 197 \pm 62 | 206 \pm 95 | 212 \pm 112 | 260 \pm 128 | 204 \pm 65 | 171 \pm 49 | 138 \pm 70 | 130 \pm 25 | 0.003 | 0.720 | 0.789 |
| Benzenemethanol | nd ^c | nd ^c | 15 \pm 26 ^{bc} | 6 \pm 6 ^c | 19 \pm 18 ^{bc} | 7 \pm 5 ^c | 27 \pm 12 ^{bc} | 22 \pm 11 ^{bc} | 82 \pm 34 ^a | 66 \pm 32 ^{ab} | <0.001 | 0.247 | 0.929 |
| Benzeneacetaldehyde | nd ^b | nd ^b | 44 \pm 50 ^{ab} | 54 \pm 62 ^{ab} | 114 \pm 104 ^{ab} | 189 \pm 191 ^{ab} | 248 \pm 227 ^{ab} | 369 \pm 371 ^a | 187 \pm 188 ^{ab} | 106 \pm 127 ^{ab} | 0.002 | 0.583 | 0.426 |
| 5-Ethylidihydro-2(3H)-furanone | nd ^b | nd ^b | 12 \pm 11 ^{ab} | nd ^b | 32 \pm 8 ^b | 3 \pm 6 ^b | 32 \pm 7 ^a | 25 \pm 5 ^{ab} | 36 \pm 18 ^a | 18 \pm 14 ^{ab} | <0.001 | <0.001 | 0.094 |
| 1-Octanol | 41 \pm 28 ^{ab} | 40 \pm 18 ^{ab} | 44 \pm 52 ^a | 12 \pm 11 ^b | 18 \pm 13 ^{ab} | 21 \pm 7 ^{ab} | 27 \pm 15 ^{ab} | 24 \pm 13 ^{ab} | 17 \pm 8 ^{ab} | 15 \pm 12 ^{ab} | 0.001 | 0.103 | 0.170 |
| Nonanal | 318 \pm 197 | 334 \pm 93 | 248 \pm 79 | 293 \pm 127 | 295 \pm 107 | 393 \pm 264 | 366 \pm 148 | 263 \pm 41 | 234 \pm 170 | 199 \pm 118 | 0.061 | 0.915 | 0.606 |
| Benzeneethanol | nd | nd | nd | nd | 8 \pm 9 | 5 \pm 3 | 66 \pm 66 | 62 \pm 55 | 53 \pm 45 | 58 \pm 46 | <0.001 | 0.979 | 0.996 |
| 5-Butylidihydro-2(3H)-furanone | nd | nd | nd | nd | 2 \pm 4 | nd | 2 \pm 2 | 2 \pm 3 | 9 \pm 10 | 2 \pm 3 | 0.011 | 0.258 | 0.286 |
| Σ | 10401 \pm 4646 | 11934 \pm 4481 | 13330 \pm 8420 | 11770 \pm 5562 | 15926 \pm 5213 | 17646 \pm 695 | 23579 \pm 7666 | 17419 \pm 3150 | 20200 \pm 5312 | 19688 \pm 9220 | 0.002 | 0.596 | 0.727 |

^A Values followed by different letters in the same row were significantly different at a level of 5%. nd: not detected.

Variations in the lipid oxidation-derived compounds agree previous researches (Antequera *et al.*, 1992; Jurado *et al.*, 2002): an increase during the first stages and a subsequent decrease during the cellar phase. This decrease in the lipid oxidation-derived compounds could be due to their participation in other chemical reactions yielding other volatile or non volatile compounds and probably to a decrease in the oxidation reactions. Results agree with those by Ruiz *et al.*, (1999) for Iberian ham, who reported a decrease in some lipid oxidation derived compounds from day 420 to day 600 of processing. Also Hinrichsen and Pedersen (1995) found for Parma hams a decrease in hexanal, pentanal and n-pentanol from day 125 to day 485 of ripening.

A previous study focused on the evolution of Iberian ham volatile compounds extracted by using a direct-extraction-SPME method that did not allow an accurate control of temperature extraction (Andrés *et al.* 2002), did not show a clear trend in the evolution of lipid oxidation-derived compounds: increases and decreases in these volatile compounds were observed during Iberian ham processing. In the present study, the extraction was performed at controlled temperature (at 35 °C during the extraction procedure), which has avoided the variability due to the extraction temperature.

3.2. Detection of differences caused by the feeding system of pigs

The effect of the feeding system (Montanera vs HO-Pienso hams) on the volatile compounds was

slight but significant for some compounds. No significant effect of the feeding system on lipid oxidation aldehydes (such as hexanal, nonanal, octanal and heptanal) was found (Table 3).

Conversely, a significant effect was observed on six volatile compounds (Table 3). Some of them were more abundant in Montanera than in Pienso hams (butanoic acid, 2,6-dimethylpyrazine and 1-octen-3-ol), and they have been described as odor-active compounds of dry-cured ham (Blank *et al.*, 2001; Carrapiso *et al.*, 2002b; Flores *et al.*, 1997). Acetaldehyde, which also have been identified as an odorant of Iberian ham (Carrapiso and García, 2007), was also affected by the feeding system, being more abundant in HO-Pienso hams than in Montanera hams. In any case, these small differences could be important because they involve compounds that are Iberian ham odorants, and in fact remarkable differences between the flavour characteristics of these Montanera and HO-Pienso hams were found in the sensory analysis (Jurado *et al.*, in press).

For some compounds results did not match with those of other studies (López *et al.*, 1992; Cava *et al.*, 2000b; Carrapiso *et al.*, 2003b). Differences in results are probably related to the different extraction techniques applied, and to other factors related to raw material (such as concentrate feeding composition, montanera feeding period length) or to the ripening conditions (temperature, relative humidity and ripening time). These factors could cause a great heterogeneity in the results.

The weak influence of the feeding system on volatile compounds could be due to the great

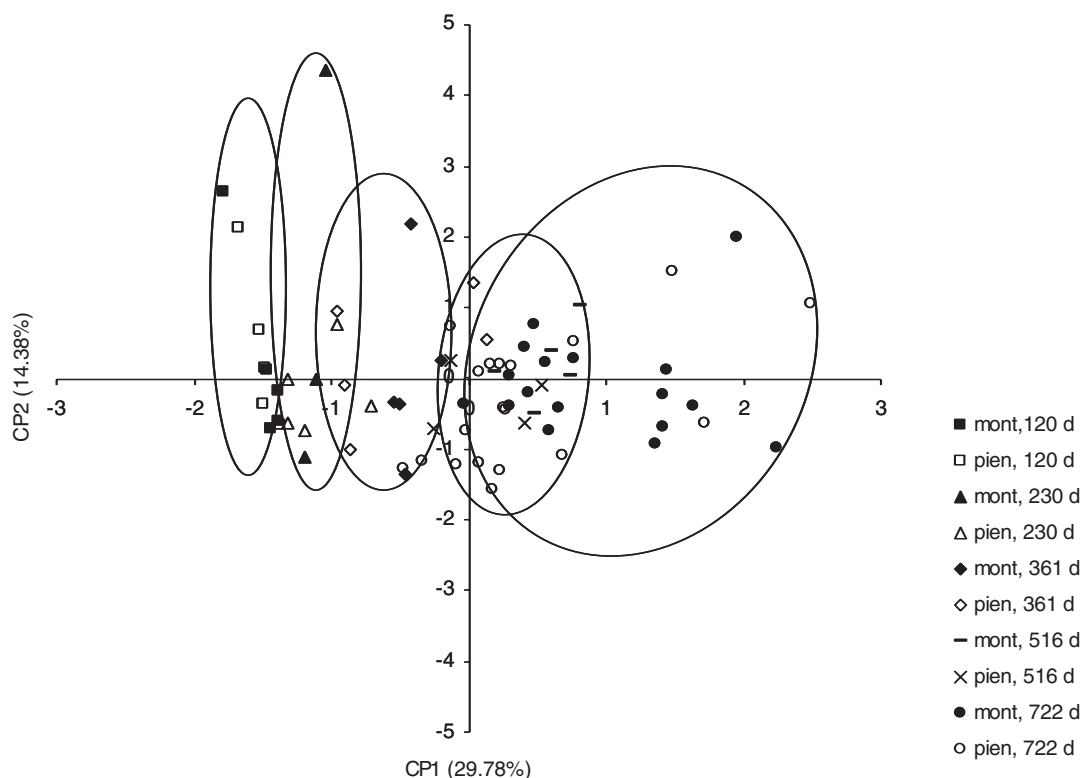


Figure 1.

Projection of the samples onto the space defined by the first two principal components (PC1 and PC2) obtained.

similarity between the fat composition of the feeding used for the pigs reared in the HO-Pienso (a high oleic acid concentrate) and in the Montanera system (grass and acorns), which led to obtain a similar intramuscular fat composition and content in the raw meat (Muriel *et al.*, 2002). Fat content has a marked effect on the release of the volatile compounds from meat products (Chevance and Farmer, 1999; Carrapiso, 2007), and also has an outstanding influence, as their fatty acid profiles does, on the generation of volatile compounds (Mottram, 1998; Elmore *et al.*, 2002; Hidalgo and Zamora, 2004).

3.3. Principal component analysis

A principal component analysis was performed to study the relationship among samples and among variables. The value for the Kaiser-Meyer-Olkin measure of sampling adequacy was suitable (0.730).

Figure 1 shows the projection of the samples onto the space defined by the two first principal components (PC1 and PC2) accounted for the 46.16% of the total variability. The PC1 axis was

mainly composed of volatile compounds coming from amino acids by means of Maillard and Strecker reactions (coefficients greater than 0.6) (Table 4). All these volatile compounds were placed in the positive zone of the PC1 axis, which indicates a positive relationship among them.

On the other hand, volatile compounds which come from oxidative processes had little loads in the PC1 for most compounds (Table 4). The largest load appeared for 1-pentanol (-0.513), revealing a negative relation between this variable and the volatile compounds coming from Strecker and Maillard reactions.

As it can be observed in the Figure 1, the samples were grouped according to the ripening time in the PC1 axis: 120 days aged samples appear in the negative semi-axis of PC1, which was characterised by low levels of Strecker and Maillard compounds (Table 4), and as the samples had a highest degree of ripening, they were nearest to the positive area of PC1, which was mainly explained by Strecker and Maillard compounds (Table 4). The samples with a ripening time of 516 and 722 days

Table 4
Factor loadings for each volatile compounds on the first two principal components obtained

| Compounds | Component (% variance explained) | |
|---------------------------------|----------------------------------|--------------|
| | CP1 (29.78%) | CP2 (14.38%) |
| Acetaldehyde | 0.477 | -0.052 |
| Metanethiol | 0.714 | -0.019 |
| 2-Propanone | -0.005 | 0.010 |
| 2-Methylpropanal | 0.797 | 0.176 |
| Acetic acid | 0.657 | 0.042 |
| 3-Methylbutanal | 0.687 | 0.138 |
| 2-Methylbutanal | 0.880 | 0.138 |
| 2-Pentanone | 0.061 | 0.071 |
| Pentanal + Heptane | 0.339 | 0.623 |
| 3-Methyl-1-butanol | 0.734 | -0.003 |
| 2-Methyl-1-butanol | 0.789 | -0.031 |
| Dimethyl disulfide | 0.688 | 0.055 |
| 1-Pentanol | -0.513 | 0.634 |
| Butanoic acid | 0.486 | 0.242 |
| 2,3- Butanodiol | 0.507 | 0.238 |
| Hexanal + Octane | -0.149 | 0.742 |
| 3-Methylbutanoic acid | 0.472 | 0.098 |
| 1-Hexanol | -0.262 | 0.664 |
| 2-Heptanone | 0.420 | 0.107 |
| Heptanal | -0.001 | 0.769 |
| 3-Methylthiopropional | 0.734 | -0.009 |
| 2,6-Dimethylpyrazine | | |
| + Dihydro 2(3H)-furanone | 0.808 | 0.063 |
| Benzaldehyde | 0.690 | 0.288 |
| 1-Octen-3-ol | -0.250 | 0.809 |
| 2-Pentylfuran | 0.174 | 0.140 |
| Octanal | -0.225 | 0.664 |
| Benzenemethanol | 0.788 | 0.028 |
| Benzeneacetaldehyde | 0.507 | 0.021 |
| 5-Ethylidihydro-2(3H)-furanone | 0.625 | 0.144 |
| 1-Octanol | -0.238 | 0.792 |
| Nonanal | -0.163 | 0.539 |
| Benzeneethanol | 0.598 | -0.098 |
| 5-Butylidihydro-2(3H)-furanone | 0.508 | 0.012 |

appear in the positive zone of the axis PC1, which shows that these samples were characterized by higher levels of Strecker and Maillard compounds and lower levels of lipid oxidation compounds. The PC2 axis was mainly composed of lipid oxidation derived compounds, but did not show any trend with regard to the effect of ripening time or pig feeding.

4. CONCLUSIONS

Ripening time had a marked effect on the volatile compounds of Iberian ham, especially on Strecker and Maillard volatile compounds whose abundances increased throughout processing. The abundance of some of these compounds (2-methylpropanal, 2-methylbutanal, 2-methyl-1-butanol, disulfide dimethyl, 2,6-dimethylpyrazine + dihydro-2(3H)furanone and benzenemethanol) could be used as indicators of the ripening time and, therefore, as flavour development indicators. Also, some odour-active compounds (butanoic acid, 2,6-dimethylpirazyne and 1-octen-3-ol) were more abundant in Montanera than in Pienso hams, and they could be used as indicators of the feeding system.

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