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# Lipid oxidative changes throughout the ripening of dry-cured Iberian hams with different salt contents and processing conditions

A.I. Andrés\*, R. Cava, J. Ventanas, E. Muriel, J. Ruiz

Food Science, School of Veterinary Sciences. University of Extremadura, Cáceres, Spain

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#### Abstract

Hexanal content and TBARs were monitored in *Semimembranosus* and *Biceps femoris* muscles throughout the ripening of 46 drycured Iberian hams processed with different amounts of salt (6% vs. 3% w/w) and different processing systems (traditional vs. modified). Rancid odour and rancid flavour were sensorially analysed in the final product. Hams processed at higher temperatures during the drying stage, following a traditional system, showed lower values in this phase for hexanal content in both muscles (P < 0.001), but TBARs showed the opposite behaviour (P < 0.001 and P < 0.05 for *Semimembranosus* and *Biceps femoris*, respectively). Salt content affected hexanal content of each muscle in a different way. *Semimembranosus* muscles from traditionally processed hams showed lower rancid flavour scores (P < 0.001) while salt did not significantly affect rancid odour or aroma scores (P > 0.05). Evidently the effects of salt content and processing temperature, within studied ranges, on lipid oxidation are limited. In fact, increased processing temperature, during the drying stage, does not lead to greater oxidation in dry-cured ham, in spite of the well known pro-oxidant effect of temperature.

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Keywords: Lipid oxidation; Hexanal; TBARs; Dry-cured ham; Salt; Processing temperature

# 1. Introduction

Lipid oxidative phenomena that take place throughout the processing of dry-cured Iberian ham lead to the formation of volatile carbonyls (Antequera et al., 1992) which strongly affect the flavour of the final product (Ruiz, Ventanas, Cava, García, Andrés, & García, 1999). Contents of volatile aldehydes from lipid oxidation in dry-cured Iberian ham are markedly higher than in other types of dry-cured hams, due to the longer processing and more extreme processing conditions (Ruiz, Muriel, & Ventanas, 2002). The potentially negative implications of this fact on the flavour of drycured Iberian ham are counteracted by the high number of volatile flavour compounds of these hams, showing many aromatic nuances (Ruiz, García, Muriel, Andrés, & Ventanas, 2002). Thus, a certain level of compounds

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with rancid notes is needed to achieve the typical flavour, but an excess in such aromatic notes leads to an overall unpleasant flavour.

The development of lipid oxidation in dry-cured meat products relies on a number of factors, such as the raw material composition and characteristics (Ruiz & Lopez-Bote, 2002), the processing conditions (Buscailhon, Berdagué, & Monin, 1993; Toldrá, Flores, & Sanz, 1997) and the amount and type of additives and ingredients added to the product (Aguirrezabal, Mateo, Domínguez, & Zumalacárregui, 2000). Salt addition is essential for (1) achieving microbiological stability (through decreasing water activity), (2) making possible the solubilization of muscle proteins which in turn allows gel formation and development of an optimum texture, and (3) for imparting salty taste in the final product (Martin, 2001).

On the other hand, salt affects, (to a certain extent) some chemical and biochemical reactions, such as proteolysis (Martín, Antequera, Córdoba, Timón, & Ventanas, 1998), lipolysis (Martín, Córdoba, Ventanas, &

<sup>\*</sup> Corresponding author. Tel.: +34-924-286-200; fax: +34-924-272-208.

E-mail address: aiandres@unex.es (A.I. Andrés).

Antequera, 1999) and lipid oxidation (Kanner, Harel, & Joffe, 1991). As far as lipid oxidation is concerned, salt has been described as a pro-oxidant agent in several studies (Coutron-Gambotti, Gandemer, Rousset, Maestrini, & Casabianca, 1999; Kanner et al., 1991). Thus, strategies aimed to reduce salt content in dry-cured ham, due to the implications of salt content in the human diet on cardiovascular health (Morgan, Aubert, & Brunner, 2001), could affect the rate and degree of lipid oxidation development in dry-cured Iberian ham.

Slight increases in temperature during the processing of dry-cured ham seem to positively influence the development of lipid oxidative reactions according to Antequera et al. (1992). However, these authors also detected a decrease in lipid oxidation markers after periods in which the temperature rose. Nevertheless, no attempt to study the influence of processing temperature, nor that of the amount of added salt, on the lipid oxidative phenomena during the processing of drycured Iberian ham has been carried out. Thus, this work was aimed to study the development of oxidative reactions during processing of dry-cured Iberian hams with different salt contents and processing conditions.

### 2. Materials and methods

## 2.1. Processing of hams and sampling

Forty-six thighs were obtained from 23 Iberian  $\times$  Duroc pigs which similar genetics that had been reared under the same rearing system. Six of them were sampled and analysed (raw ham -R-), and the rest were processed into dry-cured Iberian hams. Hams were placed on shelves in a cold room held at 1–3 °C and salted by individual addition of a controlled amount of salt in the lean part of the raw ham. Two different salt levels were considered: a group of 20 hams was salted with 6% of salt (w/w) (high salt batch) (HS), whereas the other 20 hams were salted by adding 3% of salt (w/w) (low salt batch) (LS). Salting was completed when there was no visible salt on the ham surface. After completion of salting, all hams were held at 2–5 °C and

Table 1
Sampling during the processing of the Iberian hams

85-75% relative humidity for 60 days (postsalting phase). Temperature was, thereafter, increased from 5 to 20 °C at 0.25 °C/day during 60 days, while relative humidity was progressively reduced to 65%. Before the beginning of the drying stage, HS and LS batches were divided into further two groups, each one following different processing thereafter. Half the hams of each salt level followed a process which tried to mimic the temperature evolution of the traditional processing. This group (traditional -T-) was processed at a maximum temperature of  $28 \pm 2$  °C during the drying stage (77 days), followed by a cellar phase (212 days) at  $15.5\pm0.5$  °C. The other group of hams was ripened following a modified processing (modified -M-), in which the temperature was kept constant  $(19\pm1 \ ^{\circ}C)$ during both the drying stage (77 days) and the cellar stage (137 days). Both processes took 415 days and hams were (bone-in)  $7.3\pm0.5$  kg in weight. Stages at which hams were sampled and number of samples taken are shown in Table 1. In the raw hams (R) and at the end of cellar (EC), Semimembranosus and Biceps *femoris* muscles were removed from the hams, whereas, in the rest of the stages, samples were obtained by extraction of a cylinder, sized  $10 \times 2.5$  cm, using a stainless steel tube with a cutting edge. These samples mainly involved Semimembranosus and Biceps femoris muscles, which were perfectly identifiable. Samples were vacuumpackaged and kept frozen at -80 °C until analysed.

## 2.2. Measurement of lipid oxidation

Thiobarbituric acid-reactive substances (TBARs) were measured by following the extraction method described by Buege and Aust (1978), with modifications taken from Sorensen & Jorgensen (1996). One gram of sample was homogenized with 9 ml 1.15% potassium chloride for 30 s and centrifuged at 4000 rpm for 15 min. During homogenisation, tubes were kept in ice to avoid heating. Two millitres of trichloroacetic acid-thiobarbituric acid-hydrochloric acid (15%, 0.375%, 0.25 N, respectively) reagent were added to 1 ml of the supernatant. The solution was heated at 100 °C for 40 min, cooled and again centrifuged. Absorbance was

Batches	Salting (%w/w)	Type of processing <sup>a</sup>	Number of samples in each sampling <sup>b</sup>					
			R	PS	EPS	D	ED	EC
HS-T	6	Т	6	5	5	5	5	6
HS-M	6	М	6	5	5	5	5	6
LS-T	3	Т	6	5	5	5	5	6
LS-M	3	М	6	5	5	5	5	6

<sup>a</sup> T (Traditional); M (Modified).

<sup>b</sup> R (raw ham = 0 days); PS (postsalting = 102 days); EPS (end of postsalting = 138 days); D (drying = 173 days); ED (end of drying = 203 days); EC (end of cellar = 415 days).

measured at 530 nm on a spectrophotometer (model U-2000, Hitachi, Tokio) and concentration of malonaldehyde (MDA) was calculated from a standard curve in triplicate, using solutions of 1,1,3,3-tetraethoxypropane (TEP) (Merck, Schchardt). TBARs were expressed as mg MDA kg<sup>-1</sup> muscle.

Hexanal content was quantified by headspace-solid phase microextraction (SPME) and GC/MS (gas chromatograph Hewlett-Packard 5890 series II, coupled to a mass selective detector Hewlett-Packard HP-5791 A). A 0.5 g of minced sample and 1.5 ml of distilled water were thoroughly mixed in a vial, which was then closed with a teflon/silicone septum. An SPME fibre (75 µm carboxen-polydimethylsiloxane coating) was inserted through the septum and exposed to the headspace of the vial. Extraction was carried out at 40 °C for 30 min with stirring in a water bath. After extraction, the SPME fibre was immediately transferred to the injector of the chromatograph which was in splitless mode at 280 °C. The separation of hexanal was performed on a 5% phenyl-methyl silicone (HP-5)-bonded phase fused silica capillary column (Hewlett-Packard, 50 m×0.32 mm i.d, film thickness 1.05 m, operating at 6 psi of column head pressure. Oven program was: 40 °C for 10 min, 5 °C min<sup>-1</sup> to 200 °C, and held at 200 °C for 5 min. Transfer line to the mass spectrometer was maintained at 280 °C. The mass spectra were obtained using a mass selective detector by electronic impact at 70 eV, a multiplier voltage of 1756 V and collecting data at a rate of 1 scan  $s^{-1}$ over the m/z range 30-500. Hexanal was tentatively identified by comparing mass spectra and retention time with that of a standard (Sigma, St. Louis, USA) injected under the same conditions. Hexanal content was calculated using a standard curve with solutions of hexanal (Sigma, St. Louis, USA) in triplicate, extracted and analysed under exactly the same conditions as the samples. Hexanal content was expressed as ng hexanal  $g^{-1}$ muscle.

### 2.3. Sensory analysis

Slices of *Biceps femoris* and the *Semimembranosus* muscles from each dry-cured ham were assessed by a trained panel of 12 members, using a descriptive analysis method (García, Ventanas, Antequera, Ruiz, Cava, & Alvarez, 1996; Ruiz, Ventanas, Cava, Timón, & García, 1998). Panellists were trained and had participated in sensory evaluation of dry-cured ham for 2 years. Individual flavour and aroma recognition thresholds were used to select the subjects. Selected subjects underwent further training in dry-cured ham sensory characteristics during 2 years, using hams with different characteristics (e.g. feeding, breed.). Subjects had a total of 120 h of training in preparation for descriptive analysis. Consistency of panellists was validated using the Rasch model (García et al., 1996).

Two samples from two different hams were evaluated in each session. The panel was held at 11 a.m., 3 h after breakfast. Three 1.0 mm thick slices of about 8 g each were given to the panellists. Slices were obtained using a commercial slicing machine and were served immediately on glass plates, both the slices and the plates being at room temperature (20°–23 °C). A glass of about 100 ml of water at 12 °C was provided for each assessor between the two samples. All sessions were done in a six booth sensory panel room at 22 °C equipped with white fluorescent lighting. Twenty traits of sensory characteristics of dry-cured ham were assessed on a continuous non-structured scale ranging from the lowest intensity of each trait (left side) to the highest (right side), following the sensory descriptive test developed previously by García et al. (1996). In the present study, only rancid odour and flavour were considered. The other traits are considered elsewhere. The sensory traits, their definitions and extremes have been explained elsewhere (Ruiz et al., 1998). Each assessor evaluated the samples on a computerized system for direct recording of data (Fizz, Biosystemes, France).

#### 2.4. Statistical analysis

The effect of the amount of added salt (6% vs. 3%) and processing conditions (traditional vs. modified) was analysed by a two-way analysis of variance, together with their interaction (salt  $\times$  processing), using the GLM procedure (SPSS 10.0). The Tukey's test was used at the 5% level to make comparisons between sample means when pertinent.

## 3. Results and discussion

Hexanal content (ng g<sup>-1</sup> muscle) in Semimembranosus and Biceps femoris muscles throughout the processing of dry-cured Iberian hams with different salt contents and processed under different systems is shown in Table 2. Hexanal content has been widely used for monitoring oxidative stability in meat and meat products (Shahidi, Yun, Rubin, & Wood, 1987). Hexanal levels found in this work were considerably lower than values observed by other authors in dry-cured Iberian ham (Antequera et al., 1992; Martín, Timón, Petrón, Ventanas, & Antequera, 2000). These differences could be partly due to differences in the raw matter composition and in the processing conditions. However, the different extraction methods are most likely the main cause for differences found among these works. In other studies, in which headspace-SPME was used to determine hexanal content in other meat products, similar values to those found in the present study were obtained (Elmore, Mottram, & Hierro, 2000).

Despite the group of hams and the muscle considered, hexanal content showed an increasing pattern throughout the processing, with two peaks, one at the drying stage (D) and the other one at the end of cellar (EC) (Table 2). The two-phase rising in hexanal content in dry-cured Iberian ham has been previously found in other works (Antequera et al., 1992). Such a biphasic pattern was linked to variations in the temperature during the processing. In fact, a decrease in oxidative stability and a higher production of lipid oxidationderived compounds, as a result of an increase in temperature, has been described in model systems and meat products (Farmer, 1992; Tamura, Kitta, & Shibamoto,

Table 2

Hexanal content (ng  $g^{-1}$  of muscle) (mean±standard error of the mean) of muscles Semimembranosus and Biceps femoris throughout the ripening of dry-cured Iberian hams with either 6% (HS) or 3% (LS) salt (w/w) and processed under a traditional (T) or a modified (M) system

Sampling<sup>a</sup> Batch Semimembranosus Biceps femoris  $0.19^{3/3/1/3} \pm 0.10$  $0.27^{3/2/2/3} \pm 0.07$ R  $0.59^{23/3} \pm 0.12$  $0.70^{23/2}\!\pm\!0.10$ PS HS  $0.56^{1/3} \pm 0.16$ LS  $0.75^{2/3}\!\pm\!0.11$ P (salt) ns<sup>b</sup> ns  $0.09^{3/3} \pm 0.02$  $0.07^{3/2} \pm 0.01$ EPS HS LS  $0.10^{1/3}\!\pm\!0.01$  $0.09^{2/3} \pm 0.00$ P (salt) ns ns D HS-T  $1.02b^2 \pm 0.06$  $1.47b^2 \pm 0.13$ HS-M  $7.82a^{1}\pm0.85$  $10.8a^{1}\pm2.47$  $1.95b^2 \pm 0.48$ LS-T  $1.80b^{1}\pm0.19$ LS-M  $7.70a^{1}\pm1.89$  $13.0a^{1}\pm1.98$ P (salt/processing/ ns/z/n $ns/^{z}/ns$ interaction) ED  $0.25b^3 \pm 0.04$  $1.18a^{23}\pm0.17$ HS-T HS-M  $0.76a^3 \pm 0.11$  $0.51b^2 \pm 0.13$ LS-T  $0.54b^1\!\pm\!0.16$  $0.42b^2\!\pm\!0.08$  $0.81a^{23}\!\pm\!0.08$  $0.52b^3 \pm 0.16$ LS-M P (salt/processing/ x/ns/x ns/y/ns interaction) ED  $1.54a^{1}\pm0.22$  $6.52ab^{1}\pm0.48$ HS-T HS-M  $2.63a^2 \pm 0.35$  $10.66a^{1}\pm1.15$  $3.59a^1 \pm 1.20$  $6.50ab^1 \pm 1.17$ LS-T  $5.44a^{12}\pm1.79$ LS-M  $5.75b^2 \pm 1.37$ P (salt/processing/ x/ns/ns x/ns/ns interaction)

Different letters (a-c) within the same stage mean significant differences between batches (P < 0.05). Different superscript numbers (1–3) within the same batch between different stages mean significant differences (P < 0.05).

<sup>a</sup> R (raw ham = 0 days); PS (postsalting = 102 days); EPS (end of postsalting = 138 days); D (drying = 173 days); ED (end of drying = 203 days); EC (end of cellar = 415 days)

<sup>b</sup> ns, P > 0.05.

- $^{x}$  P < 0.05.
- $^{y} P < 0.01.$
- $^{z}$  P < 0.001.

1991). In French and Italian hams, increasing hexanal levels during the processing were also observed (Buscailhon et al., 1993; Hindrichsen & Pedersen, 1995), though this two-phase oxidative trend was not evident, most likely due to the shorter processing times of these types of hams.

Salt content significantly (P < 0.05) affected hexanal content of Semimembranosus muscle at the end of cellar (EC) stage, HS hams showing lower values than LS ones. On the other hand, HS hams showed significantly (P < 0.05) higher hexanal content values in Biceps femoris muscle at the end of drying (ED) and end of cellar (EC) samplings. Such different behaviour, the

Table 3

Malondialdehyde content (mg kg<sup>-1</sup> of muscle)(mean±standard error of the mean) of muscles Semimembranosus and Biceps femoris throughout the ripening of dry-cured Iberian hams with either 6% (HS) or 3% (LS) salt (w/w) and processed by a traditional (T) or a modified (M) system

Sampling <sup>a</sup>	Batch	Semimembranosus	Biceps femoris	
R		$0.08^{3/3/3/3} \pm 0.00$	$0.09^{2/2/23/3} \pm 0.02$	
PS	HS	$0.13^{3/23} \pm 0.08$	$0.12^{2/2} \pm 0.02$	
	LS	$0.12^{23/3} \pm 0.21$	$0.08^{3/3} \pm 0.02$	
	P (salt)	ns <sup>b</sup>	ns	
EPS	HS	$0.15^{3/23} \pm 0.02$	$0.16^{2/2} \pm 0.00$	
	LS	$0.18^{23/23}\!\pm\!0.02$	$0.20^{23/23}\!\pm\!0.02$	
	P (salt)	ns	ns	
D	HS-T	$0.59a^{1}\pm0.01$	$0.35a^{1}\pm0.04$	
	HS-M	$0.21c^{23}\pm0.01$	$0.25ab^{1}\pm0.02$	
	LS-T	$0.35b^1 \pm 0.03$	$0.24b^2 \pm 0.03$	
	LS-M	$0.24c^2 \pm 0.02$	$0.20b^{23}\pm0.03$	
	P (salt/processing/ interaction)	z/z/z	<sup>x</sup> / <sup>x</sup> /ns	
ED	HS-T	$0.19a^3 \pm 0.03$	$0.16b^2 \pm 0.02$	
	HS-M	$0.27a^2 \pm 0.03$	$0.24a^2 \pm 0.02$	
	LS-T	$0.18a^2 \pm 0.02$	$0.14b^{123}\pm0.01$	
	LS-M	$0.26a^2 \pm 0.02$	$0.27a^{12} \pm 0.02$	
	P (salt/processing/ interaction)	ns/ <sup>y</sup> /ns	ns/ <sup>z</sup> /ns	
ED	HS-T	$0.40^2 \pm 0.07$	$0.48^{1}\pm0.06$	
	HS-M	$0.52^{1}\pm0.08$	$0.45^{1}\pm0.07$	
	LS-T	$0.34^{1}\pm0.03$	$0.38^{1}\pm0.04$	
	LS-M	$0.39^{1}\pm0.04$	$0.40^{1} \pm 0.05$	
	P (salt/processing/ interaction)	ns/ns/ns	ns/ns/ns	

Different letters (a-c) within the same stage mean significant differences between batches (P < 0.05). Different superscript numbers (1–3) within the same batch between different stages mean significant differences (P < 0.05).

<sup>a</sup> R (raw ham = 0 days); PS (postsalting = 102 days); EPS (end of postsalting=138 days); D (drying=173 days); ED (end of drying = 203 days); EC (end of cellar = 415 days)

<sup>b</sup> ns, P > 0.05.

 $^{x} P < 0.05.$ 

- $^{y} P < 0.01.$
- $^{z}$  P < 0.001.

small differences found and the lack of differences at most stages underline the scarce influence of salt content on lipid oxidative changes in dry-cured Iberian ham at the levels tested in this study. The different behaviour in each muscle could be related to the pattern of salt diffusion through the whole ham, as well as other characteristics, such as fat content, fat composition, balance of antioxidants/pro-oxidants or enzymatic activity (Andrés, Cava, Mayoral, Tejeda, Morcuende, & Ruiz, 2001; Lauridsen, Nielsen, Henckel, & Sorensen, 1999). Biceps femoris is an internal muscle, covered with a thick layer of subcutaneous fat on one side, and with several muscles, fascias and intermuscular fat on the other. Thus, salt content in this muscle slowly rises throughout the processing, and is not high enough to cause effects until a long time after salting. On the other hand, Semimembranosus is close to the surface, with no subcutaneous fat on it, and thus, salt content rapidly rises after salting.

Processing markedly influenced hexanal content at the drying stage (D) in both muscles (P < 0.001) and at the end of the drying stage (ED) of Semimembranosus (P < 0.01), those hams processed under a modified (M) system showing higher values in all cases. This result is strange since, during the drying stage, hams processed under the modified system were kept at a constant temperature of 19 °C while, in the traditional system, the temperature was progressively raised to 28 °C. Evidently lipid oxidation during the processing of drycured ham is not directly dependent on the processing temperature. The amount of compounds from lipid oxidation depends on their rate of formation and degradation or reaction with other compounds. Therefore, the higher level of hexanal at the drying stage in those hams processed at lower temperatures, is most likely due to a lower rate of reaction with other compounds. Other authors have detected the development of Maillard reactions between compounds from lipid oxidation and free amino acids even at room temperature (Pripis-Nicolau, de Revel, Bertrand, & Maujean, 2000). In fact, such type of reactions has been previously described in dry-cured Iberian ham (Ventanas et al., 1992). On the other hand, temperature positively influences proteolytic enzyme activity, and thus, free amino acid content (Arnau, Guerrero, & Gou, 1997; Córdoba, Antequera, García, Ventanas, & López-Bote, 1994). Greater amounts of free amino acids in those hams processed at higher temperatures could therefore be a reason for the lower amounts of hexanal.

Processing conditions did not significantly affect hexanal content in remaining stages, although hams processed in a modified system tended to show higher values in the final product (EC stage).

TBARs in *Semimembranosus* and *Biceps femoris* muscles of dry-cured Iberian hams, processed with different amount of salt and different ripening conditions,

are shown in Table 3. TBARs also followed a biphasic type of behaviour, with marked increases at the drying stage (D) and the end of the cellar stage (EC), agreeing with the pattern described by other authors (Antequera et al., 1992). Cava, Ruiz, Ventanas, and Antequera (1999) also found a marked increase during the drying stage, with similar values to those observed in the present study.

Salt content significantly affected TBARs at the drying stage (D) of Semimembranosus (P < 0.001) and Biceps femoris (P < 0.05) muscles, those hams salted with a 6% w/w salt (HS) showing higher values in both muscles. Salt content did not affect TBARs values at any other stage, although those hams processed with a lower amount of salt tended to show lower TBARs in the final product (EC stage). The light pro-oxidant effect of salt detected in the TBARs agrees only in part, and at different stages, with hexanal results. This could be due to the different origins of malonaldehyde and hexanal (oxidation of 3 or 4 double-bonded fatty acids and oxidation of linoleic acid, respectively), together with different rates of formation and further reactions of both compounds with other substances (Draper, McGirr, & Hadley, 1986; Guillén-Sans & Guzmán-Chozas, 1998). Despite these differences, it seems that the pro-oxidant effect of salt is very small in dry-cured ham, at least in the amounts considered in the present study. Several authors have detected a pro-oxidant effect of sodium chloride in model systems (Sárraga & García-Regueiro, 1998) and meat products (Aguirrezabal et al., 2000; Kanner et al., 1991). However, some others have found little or no effect of sodium chloride.

Table 4

Sensory scores for rancid odour and rancid flavour (mean±standard error of the mean) of muscles *Semimembranosus* and *Biceps femoris* from dry-cured Iberian hams with either 6% (HS) or 3% (LS) salt (w/w) and processed by a traditional (T) or a modified (M) system

	Semimembranosus	Biceps femoris
Rancid odour		
HS-T	$1.3 \pm 0.1$	$1.3 \pm 0.1$
HS-M	$1.2 \pm 0.1$	$1.4 \pm 0.2$
LS-T	$1.2 \pm 0.1$	$1.3 \pm 0.1$
LS-M	$1.3 \pm 0.1$	$1.2 \pm 0.1$
P (salt/processing/interaction)	<sup>a</sup> /ns/ns	ns/ns/ns
Rancid flavour		
HS-T	$1.3 \pm 0.1$	$1.4 \pm 0.1$
HS-M	$1.7 \pm 0.1$	$1.6 \pm 0.3$
LS-T	$1.2 \pm 0.1$	$1.4 \pm 0.1$
LS-M	$1.6 \pm 0.1$	$1.5 \pm 0.2$
P (salt/processing/interaction)	ns/z/ns	ns/ns/ns

Different letters (a–c) within the same stage mean significant differences between batches (P < 0.05). Different superscript numbers (1–3) within the same batch between different stages mean significant differences (P < 0.05).

<sup>a</sup> ns, P > 0.05.

 $^{z}$  P < 0.001.

Sárraga and García-Regueiro (1998) observed that 1– 3% salt concentration produced lower TBARs than non-salted samples, whereas 5% salt content produced an oxidative activity. The scarce pro-oxidant effect of salt in dry-cured Iberian ham agrees with sensorial results obtained in this work, since salt did not significantly influence rancid odour and rancid aroma (P > 0.05)(Table 4). Similarly, Coutron-Gambotti et al., (1999) found that higher salt content did not increase rancidity.

Processing temperature significantly affected TBARs of Semimembranosus muscle at the drying stage (D) (P < 0.001) and end of drying stage (ED) (P < 0.01). In the former stage, those hams processed in a modified (M) system showed lower values, whereas, in the latter, these hams showed higher values. Exactly the same pattern was detected in Biceps femoris muscle, those hams processed under a modified system showing significantly lower (P < 0.05) values at the drying stage (D), and significantly higher (P < 0.001) at the end of the drying (ED). Such behaviour did not agree with that found for the hexanal content, reinforcing differences between both measurements cited previously. Regardless of these differences, it seems that the processing temperature affected the dynamics of malonaldehyde formation throughout the ripening, but not the final levels of TBARs. Therefore it seems that processing temperature scarcely influence rancidity in dry-cured ham (did not affect hexanal content or TBARs). However, sensory scores for rancid flavour showed a significant influence of processing system in the Semimembranosus muscle (P < 0.001), those hams processed in a modified system, with constant temperatures during the drying stage, showing higher rancid flavour scores. Similar behaviour was observed in the Biceps femoris muscle, though not to a significant extent. Therefore, neither hexanal content nor TBARs seems to be a good marker for rancidity in dry-cured ham. This could be due to the great number of different aromatic nuances in this product.

Sensory results underline the suitability of processing systems with increases in the temperature during the drying phase for reducing rancid flavour notes. This result is strange, since temperature positively influences lipid oxidation. Such an effect on rancidity is most probably due to its positive effect on some other reactions, such as proteolysis and Maillard reactions. This is important, since rancid notes in dry-cured Iberian ham have been negatively related to acceptability (Ruiz, García, Muriel, Andrés & Ventanas, 2002).

#### 4. Conclusion

The effect of salt content and processing temperature, within common ranges found in the processing industry,

on oxidative parameters is limited. Increasing processing temperature during the drying stage does not lead to higher levels of hexanal in dry-cured Iberian ham, which is unusual since temperature has a proven prooxidant effect.

#### References

- Aguirrezabal, M. M., Mateo, J., Domínguez, J. M., & Zumalacárregui, J. M. (2000). The effect of páprika, garlic and salt on rancidity in dry sausages. *Meat Science*, 54, 77–81.
- Andrés, A. I., Cava, R., Mayoral, A. I., Tejeda, J. F., Morcuende, D., & Ruiz, J. (2001). Oxidative stability and fatty acid composition of pig muscles as affected by rearing system, crossbreeding and metabolic type of muscle fibre. *Meat Science*, 59, 39–47.
- Antequera, T., López-Bote, J. J., García, C., Asensio, M. A., Ventanas, J., García-Regueiro, J. A., & Díaz, I. (1992). Lipid oxidative changes in the processing of Iberian pig hams. *Food Chemistry*, 45, 105–110.
- Arnau, J., Guerrero, L., & Gou, P. (1997). Effects of temperature during the last month of ageing and of salting time on dry-cured ham aged for six months. J. Sci. Food Agric., 74, 193–198.
- Buege, J. A., & Aust, S. D. (1978). Microsomal lipid peroxidation. In L.Parker S. Fleischer (Ed.), *Methods in enzymology* (pp. 302–312). New York: Academic Press.
- Buscailhon, S., Berdagué, J. L., & Monin, G. (1993). Time-related changes in volatiles compounds of lean tissue during processing of french dry-cured ham. J. Sci. Agric., 63, 69–75.
- Cava, R., Ruiz, J., Ventanas, J., & Antequera, T. (1999). Oxidative and lipolytic changes during ripening of Iberian hams as affected by feeding regime: extensive feeding and alphatocopherol acetate supplementation. *Meat Science*, 52, 165–172.
- Cordoba, J. J., Antequera, T., García, C., Ventanas, J., & López-Bote, C. (1994). Evolution of free amino acids and amines during ripening of Iberian ham. *Meat Science*, 37, 217–227.
- Coutron-Gambotti, C., Gandemer, G., Rousset, S., Maestrini, O., & Casabianca, F. (1999). Reducing salt content of dry-cured ham: effect on lipid composition and sensory attributes. *Food Chemistry*, *64*, 13–19.
- Draper, H. H., McGirr, L. G., & Hadley, M. (1986). The metabolism of malonaldehyde. *Lipids*, 21, 305–308.
- Elmore, J. S., Mottram, D. S., & Hierro, E. (2000). Two-fibre solidphase microextraction combined with gas chromatography-mass spectrometry for the analysis of volatile aroma compounds in cooked pork. J. Chromatography A, 605, 233–240.
- Farmer, L. J. (1992). Meat Flavour. In D. A. Ledward, D. E. Johnston, & M. K. Knight (Eds.), *The chemistry of muscle-based foods* (pp. 169–183). Cambridge: Royal Society of Chemistry.
- García, C., Ventanas, J., Antequera, T., Ruiz, J., Cava, R., & Alvarez, P. (1996). Measuring sensorial quality of Iberian ham by Rasch model. J. Food Qual., 19, 397–412.
- Guillén-Sans, R., & Guzmán-Chozas, M. (1998). The Thiobarbituric acid reaction in foods: a review. *Crit. Rev. Food Sci. Nut.*, 38(4), 315–330.
- Hinrichsen, L. L., & Pedersen, S. B. (1995). Relationship among flavor, volatile compounds, chemical changes, and microflora in italian-type dry-cured ham curing processing. J. Agric. Food Chem., 43, 2932–2940.
- Kanner, J., Harel, S., & Joffe, R. (1991). Lipid peroxidation of muscle food as affected by NaCl. J. Agric. Food Chem., 39, 1017–1024.
- Lauridsen, C., Nielsen, J. H., Henckel, P., & Sorensen, M. T. (1999). Antioxidative and oxidative status in muscles of pigs fed rapeseed oil, vitamin E, and copper. J. Animal Sci., 77, 105–115.

- Martin, M. (2001). Meat Curing Technology. In Y. H. Hui, W.-K. Nip, R. W. Rogers, & A. O. Young (Eds.), *Meat science and applications* (pp. 491–508). New York: Marcel Dekker.
- Martín, L., Antequera, T., Córdoba, J. J., Timón, M. L., & Ventanas, J. (1998). Effects of salt and temperature in proteolysis during ripening of Iberian ham. *Meat Science*, 49(2), 145–153.
- Martín, L., Córdoba, J. J., Ventanas, J., & Antequera, T. (1999). Changes in intramuscular lipids during ripening of Iberian drycured ham. *Meat Science*, 51, 129–134.
- Martín, L., Timón, M. L., Petrón, M. J., Ventanas, J., & Antequera, T. (2000). Evolution of volatile aldehydes in Iberian ham matured under different processing conditions. *Meat Science*, 54, 333–337.
- Morgan, T., Aubert, J-F., & Brunner, H. (2001). Interaction between sodium intake, angiotensin II, and blood pressure as a cause of cardiac hypertrophy. Am. J. Hypertension, 14(9), 914–920.
- Pripis-Nicolau, L., de Revel, G., Bertrand, A., & Maujean, A. (2000). Formation of flavor components by the reaction of amino acid and carbonyl compounds in mild conditions. J. Agric Food Chem., 48, 3761–3769.
- Ruiz, J., García, C., Muriel, E., Andrés, A. I., & Ventanas, J. (2002). Influence of sensory characteristics on the acceptability of dry-cured ham. *Meat Science*, 61, 347–354.
- Ruiz, J. & Lopez-Bote, C. (2002). Improvement of dry-cured ham quality by lipid modification through dietary means. In F. Toldrá (Ed.), *Research advances in the quality of meat and meat products*. (pp. 255–271). Trivandrum, India: Research Signpost.
- Ruiz, J. Muriel, E., & Ventanas, J. (2002). The flavour of Iberian ham.

In F. Toldrá (Ed.), *Research advances in the quality of meat and meat products*. (pp. 289–309). Trivandrum, India: Research Signpost.

- Ruiz, J., Ventanas, J., Cava, R., García, Andrés, & A.I. García, C. (1999). Volatile compounds of dry-cured Iberian ham as affected by the length of the curing process. *Meat Science*, 52, 19–27.
- Ruiz, J., Ventanas, J., Cava, R., Timón, M.L, & García, C. (1998). Sensory characteristics of Iberian ham: influence of processing time and slice location. *Food Res. Int.*, 31(1), 53–58.
- Sarraga, C., & Garcia-Regueiro, J. A. (1998). Oxidation of microsomal fraction in the processing conditions of dry-cured ham. The in-vitro effect of brine. *Meat Sci.*, 50, 295–302.
- Shahidi, F., Yun, J., Rubin, L. J., & Wood, D. F. (1987). The hexanal content as an indicator of oxidative stability and flavour acceptability in cooked ground pork. *Canadian Institute of Food Sci Tech.*, 20, 104–106.
- Sorensen, G., & Jorgensen, S. S. (1996). A critical examination of some experimental variables in the 2-thiobarbituric acid (TBA) test for lipid oxidation in meat products. *Zeitschrift für Lebensmittel-Untersuchung und Forschung*, 202, 205–210.
- Tamura, H., Kitta, K., & Shibamoto, T. (1991). Formation of reactive aldehydes from fatty acids in a Fe-2-+/H2O2 oxidation system. J. Agric. Food Chem., 39, 439–442.
- Toldrá, F., Flores, M., & Sanz, Y. (1997). Dry-cured flavour: enzymatic generation and process influence. *Food Chemistry*, 59, 523–530.
- Ventanas, J., Córdoba, J. J., Antequera, T., García, C., López-Bote, C., & Asensio, M. A. (1992). Hydrolisis and Maillard reactions during ripening of Iberian ham. J. Food Science, 57(4), 813–815.