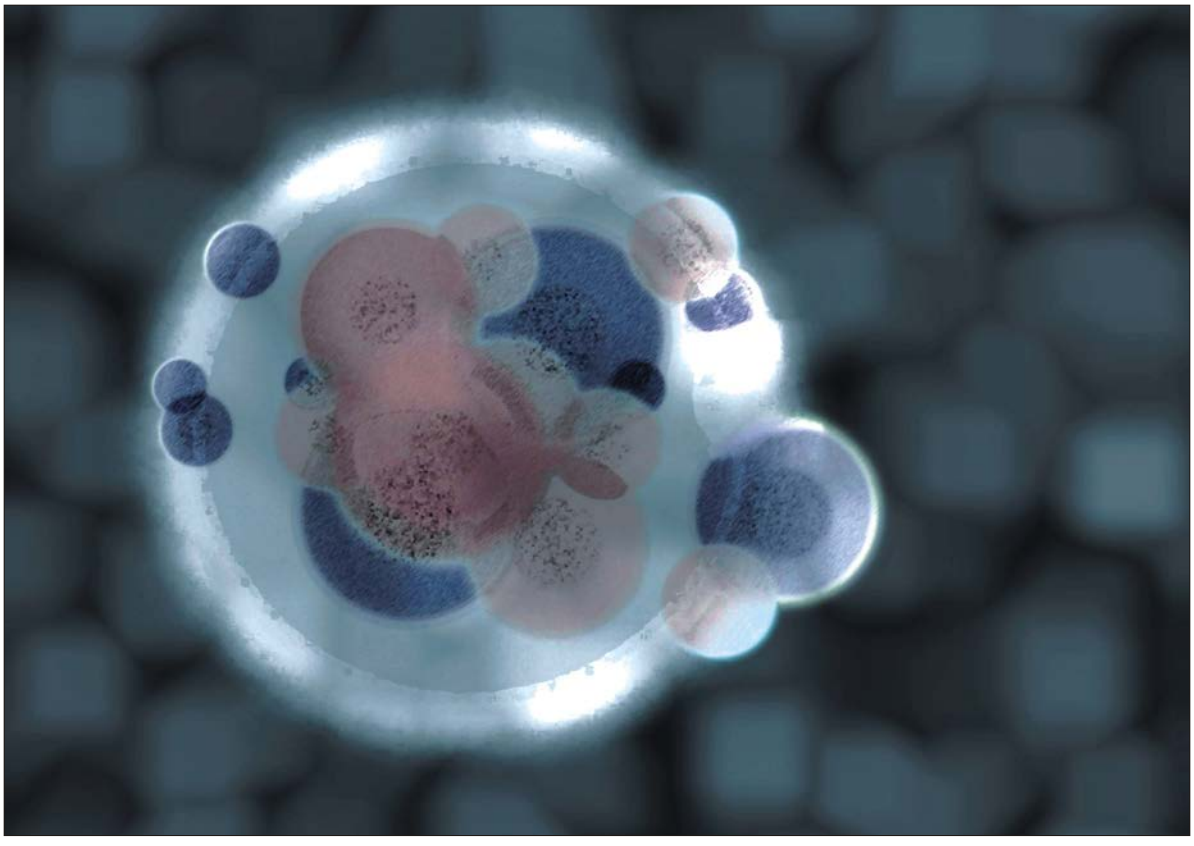

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Changes on Physico-chemical, Textural, Proteolysis, Lipolysis and Volatile Compounds During the Manufacture of Dry-cured "Lacón" from Celta Pig Breed

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Abstract: The changes in physico-chemical, textural, proteolysis, lipolysis and volatile compounds during the manufacture of dry-cured Celta "lacón" were studied. The pH value increased during the final stages of processing. While gradually declined over the curing period, TBAR´S values and hardness increased with processing time. The colour parameters, L* (from 38.68 to 35.13), a* (from 19.10 to 14.55) and b* (from 10.05 to 7.67) decreased as processing time increased. In the Free Fatty Acid (FFA) fraction, Saturated Fatty Acid (SFA) showed the highest values at the end of process, while Monounsaturated (MUFA) and Polyunsaturated Fatty Acids (PUFA) presented similar amount. Regarding, Free Amino Acids (FAA), a significant increase ($p<0.001$) from raw pieces (688.6 mg/100 g of dry matter) to the end of dry-ripening (3309.9 mg/100 g of dry matter) was observed. At the end of process the most abundant FAA detected were arginine, followed by taurine, glutamic acid and alanine, which were up to 270 mg/100 g of TS. Finally, sixty four volatile compounds were identified during the manufacture of dry-cured Celta "lacón". At the end of process, esters had become the dominant chemical compounds followed by aliphatic hydrocarbons and branched hydrocarbons.

Key words: Dry-cured meat product, physico-chemical properties, free fatty acid, free amino acid, volatile compounds

INTRODUCTION

The Celta pig breed raised in Galicia (NW Spain) until the middle of the 20th century, at which time it was substituted for commercial crossbreeds due to their higher productive capacity, prompted an important recession in this autochthonous pig breed. With the help of Regional Government of Galicia and National Government of Spain programs, the Celta pig breed has undergo a recovery in the last few years, dropping from 2.714 animals at the end of 2008 to 4.476 animals at the end of 2011 (MAGRAMA, 2012).

Nowadays, demand for autochthonous pig breeds products has increased, which is attributed to a revaluation of traditional, high-quality products. Therefore, the only option of survival of these breeds is relating to producing products with high added value. The Celta pig breed is characterized by a great rusticity that allows a perfect ability to adapt to the habitat conditions of the autochthonous forests. Among these last products we can find the "Lacón Gallego" which is manufactured from the foreleg of the pig, following a technology similar to that used for raw-cured ham and recognized as a Geographically Protected Identity (The Commission of the European Communities, 2001).

Quality in meat products have been defined as the total degree of satisfaction which the meat gives to the consumer (Jul and Zeuthen, 1981). On the other hand, the quality of dry-cured meat products is detected by the raw pieces and the manufacture process (Arnau *et al*., 2009). Some specific characteristics on fresh meat have been related with the quality of these products (Ruiz-Carrascal *et al*., 2000; Ruiz-Ramirez *et al*., 2006) like that the pig genotype (Armero *et al*., 2002; Garcia-Rey *et al*., 2004). Many parameters have been assessed to characterize dry-cured meat products such as color, texture, chemical composition (moisture, fat and protein content) and volatile compounds profile, which affects the quality of final product. Various studies have been carried out with the aim of improving the quality of dry cured lacón product. They have concerned the microbiological and biochemical (Lorenzo *et al*., 2007a, b; Lorenzo *et al*., 2008a, b; Garrido *et al*., 2012), sensorial properties (Purrinos *et al*., 2011b) and volatile compounds (Purrinos *et al*., 2011b) changes that take place during manufacture of the product. However any of

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these studies were carried out on pieces of Celta breed pig. Thus, the aim of this study was to determine the physico-chemical properties, lipolytic and proteolytic changes and volatile profile of dry-cured "lacón" from Celta pig breed.

MATERIALS AND METHODS

Experimental design and animal management: Ten pigs from the Celta breed (Barcina line) were used. All specimens, registered in the Record of Births of Stud-Book were obtained from ASOPORCEL. All animals were reared in a single group in an extensive system. They were fed *ad libitum* with commercial concentrate suited to the nutritive needs of the animals. Table 1 shows the chemical composition and fatty acid profile of the commercial feed. The animals were slaughtered at 12 months. The day before slaughter, the animals were weighed and transported to the abattoir trying to minimize the stress of the animals. Pigs were slaughtered in an accredited abattoir, using carbon dioxide to stun the animals (Lugo, Spain).

Samples: After the refrigeration period (24 h at 4°C), "lacón" samples were extracted. Fresh pieces of 4.35±0.09 kg were used. Raw pieces were salted with an excess of coarse salt. A heap was formed consisting of alternating layers of "lacón" pieces and layers of salt. In this way, the pieces were totally covered with salt and pieces remained in the pile for four days (a day per kg of weight), the temperature of the salting room was in the range 2-5°C and 80-90% relative humidity. After the salting stage, the pieces were taken from the pile, brushed, washed to remove salt excess and transferred to a post-salting chamber where they stayed for 14 days at 2-5°C and 85-90% relative humidity. After the post-salting stage the pieces were transferred to a room at 12°C and 74-78% relative humidity where drying-ripening took place for 84 days. The air convection in the drying room was intermittent and the air velocity around the pieces when the fan was running ranged between 0.3 and 0.6 m/s.

"Lacón" samples were taken from the fresh pieces, after the end of the salting stage, after 14 days of post-salting and after 84 days of drying-ripening. Each sample consisted of one whole "lacón" piece. In each sample point a total of five "lacón" samples were analyzed. Samples were transported to the laboratory under refrigerated conditions (<4°C) and analysed at this point. Once in the laboratory, the entire pieces were skinned, deboned and *Triceps brachii* muscle was extracted. The samples were stored at -80°C for no longer than four weeks until analysis.

Table 1: Chemical composition and fatty acid profile of the commercial feed

Chemical composition	Values
Moisture (%)	10.68
Crude protein (%)	13.34
Ash (%)	5.35
Fat (%)	5.55
Cellulose	4.1
Phosphate (%)	0.41
Ca (%)	1.04
Cu (mg kg^{-1})	21
Fatty acid profile (%)	
C14:0	0.74
C16:0	19.69
C16:1	1.40
C17:0	0.21
C17:1	0.18
C18:0	6.10
C18:1n9c	36.94
C18:2n6c	30.85
C20:0	0.22
C20:1	0.92
C18:3n3	2.37
C20:2	0.28
C20:5n3	0.11
SFA	26.96
MUFA	39.44
PUFA	33.60
P/S	1.24
n-6/n-3	12.47

The concentrate was formulated using the following ingredients (%): 40 wheat, 25.5% barley, 15% soybean flour, 14.6% coru, 1.5% soybean oil, 2% calcium carbonate, 1% dicalcium phosphate and 0.20% sodium chloride

Analytical methods

Reagents: Fatty acid methyl esters (FAME´s) standard mixtures and nonadecanoic acid methyl ester were acquired from Supelco Inc. (Bellefonte, PA, USA). Analytical grade and liquid chromatographic grade chemicals were purchased from Merck Biosciences (Darmstadt, Germany). Boron trifluoride (14% solution in methanol) was obtained from Panreac (Castellar del Vallès, Barcelona, Spain). AccQ.Fluor reagent kit (AQC, borate buffer) and AccQ.Tag Eluent A concentrate were acquired from Waters (Milford, MA, USA). Acetonitrile (MeCN), disodium ethylenediaminetetraacetic acid (EDTA), phosphoric acid, sodium acetate trihydrate and sodium azide were from Baker (Phillipsburg, PA, USA); triethylamine (TEA) was purchased from Aldrich (Milwaukee, WI, USA). Amino acid standards, taurine and hydroxyproline were from Sigma (St. Louis, MO, USA).

pH, Water Activity, TBAR´S Values and Colour Parameters: The pH of samples was measured using a digital pH-meter (Thermo Orion 710 A+, Cambridgeshire, UK) equipped with a penetration probe. Colour measurements were carried out using a CR-600 colorimeter (Minolta Chroma Meter Measuring Head, Osaka, Japan). Each sausage was cut and the colour of the slices was measured three times for each analytical point. CIELAB space (CIE, 1976): lightness, (L*); redness, (a*);

yellowness, (b*) were obtained. Before each series of measurements, the instrument was calibrated using a white ceramic tile. Lipid oxidation was assessed in triplicate by the 2-thiobarbituric acid (TBAR´S) method of Vyncke (1975) with the modification that samples were incubated at 96°C in a forced oven (Memmert UFP 600, Schwabach, Germany). Thiobarbituric acid reactive substances (TBAR´S) values were calculated from a standard curve of malonaldehyde (MDA) and expressed as mg MDA/kg sample. Water activity was determinated using a Fast-lab (Gbx, Romans sur Isére Cédex, France) water activity meter, previously calibrated with sodium chloride and potassium sulphate.

Chemical composition: Moisture, fat and protein (Kjeldahl N×6.25) and ash were quantified according to the ISO recommended standards 1442:1997 (ISO, 1997), 1443:1973 (ISO, 1973) and 937:1978 (ISO, 1978), respectively. Total chlorides were quantified according to the Carpentier-Vohlard official method.

WHC and texture analysis: Samples were cooked placing vacuum package bags in a water bath with automatic temperature control (JP Selecta, Precisdg, Barcelona, Spain) until they reached an internal temperature of 70°C, controlled by thermocouples type K (Comark, PK23M, UK), connected to a data logger (Comark Dilligence EVG, N3014, UK). After cooking, samples were cooled in a circulatory water bath set at 18°C during a period of 30 min. Cooking loss was calculated by measuring the differences in weight between the cooked and raw samples as follows:

$$CL = \frac{(\text{weight loss})}{(\text{initial fresh meat weight})} \times 100$$

Seven meat pieces of 1×1×2.5 cm (height x width x length) were removed parallel to the muscle fibre direction at a cross head speed of 3.33 mm/sec in a texture Analyzer (TA.XT.plus of Stable Micro Systems, Vienna Court, UK) and were completely cut using a Warner-Braztler (WB) shear blade with a triangular slot cutting edge (1 mm of thickness). Maximum shear force, shear firmness and total necessary work performed to cut the sample were obtained. Texture Profile Analysis (TPA) was measured by compressing to 60% with a compression probe of 19.85 cm^2 of surface contact. Force-time curves were recorded at a cross head speed of 3.33 mm/s and recording speed was also 3.33 mm/s. Hardness (kg), cohesiveness, springiness (mm), gumminess (kg) and chewiness (kg*mm) were obtained. These parameters were obtained using the available computer software.

Free fatty acid: Total intramuscular lipids were extracted from 5 g of ground meat sample, according to Folch *et al.* (1957) procedure. Free fatty acids were separated using NH_2-aminopropyl mini-columns as described by Garcia-Regueiro *et al.* (1994). Fifty milligrams of the extracted lipids were transesterified with a solution of boron trifluoride (14%) in methanol, as described by Carreau and Dubacq (1978) and the FAME´s were stored at -80°C until chromatographic analysis.

Separation and quantification of FAME's was carried out using a gas chromatograph, GC-Agilent 6890N (Agilent Technologies Spain, S.L., Madrid, Spain) equipped with a flame ionization detector and an automatic sample injector HP 7683 and using a Supelco SPTM-2560 fused silica capillary column (100 m, 0.25 mm i.d., 0.2 μm film thickness, Supelco Inc, Bellafonte, PA, USA). Chromatographic conditions were as follows: initial oven temperature of 120°C (held for 5 min) , first ramp at 2°C/min to 170°C (held for 15 min), second ramp at 5°C/min to 200°C (held for 5 min) and third ramp at 2°C/min to final temperature of 235°C (held for 10 min). The injector and detector were maintained at 260 and 280°C respectively. Helium was used as carrier gas at a constant flow-rate of 1.1 mL min^{-1}, with the column head pressure set at 35.56 psi. 1 μL of solution was injected in split mode (1:50). The fatty acids were quantified using nonadecanoic acid methyl ester at 0.3 mg mL^{-1}, as internal standard, was added to samples prior to fat extraction and methylation. Identification of fatty acids was performed by comparison of the retention times with those of known fatty acids and the results expressed as mg g^{-1} of fat.

Free amino acids: The extraction of free amino acids was performed, as described by Alonso *et al.* (1994). The identification and quantification of amino acids were carried out used a HPLC Alliance 2695 model (Waters, Milford, USA) and 2475 scanning fluorescence detector (Waters Milford, USA). Empower 2™ advanced software (Waters, Milford, USA) was used to control system operation and results management.

The derivatization of standards and samples and chromatographic analysis conditions were as follow: 10 μL of sample was buffered to pH 8.8 (AccQ.Flour borate buffer) to yield a total volume of 100 μL. Derivatization was initiated by the addition of 20 μL of AccQ-Fluor reagent (3 mg mL^{-1} in acetonitrile). Reaction of the AQC with all primary and secondary amines was rapid and excess reagent was hydrolyzed within 1 min. Completion of hydrolysis of any tyrosine phenol modification was accelerated by heating for 10 min at 55°C. Separations were carried out using a Water AccQ-Tag column (3.9 mm×150 mm with a 4 μm of particle size) with a flow-

ate of 1.0 mL min^{-1} and performed at 37°C. The gradient profile and composition of the mobile phase was adapted from methodology developed by Van Wandelen and Cohen, 1997. Detection was accomplished by fluorescence with excitation at 250 nm and emission at 395 nm. Amino acids were identified by retention time using an amino acid standard.

Analysis of volatile compounds: Samples were ground in a domestic blender and 10 g weighed, put into a dynamic headspace vial. The volatile compounds were extracted and concentrated in a purge and trap concentrator coupled with a cryofocusing module (Teledyne Tekmar, Mason, OH, USA).

Dynamic headspace volatile concentration: Samples were transferred into headspace vials and concentrated in a purge-and-trap concentrator (Stratum, Teledyne Tekmar, Mason, OH, USA) equipped with a cryofocusing module connected to an autosampler (Solatek 72 Multimatrix Vial Autosampler, Teledyne Tekmar, Mason, OH, USA). The sample was maintained at 60°C for 5 min and then flushed with helium at a flow rate of 60 mL min^{-1} for 20 min. Volatile compounds were adsorbed on a Tenax Trap (Strat trap, 30.48 cm, Agilent Technologies Spain, S.L., Madrid, Spain) and subsequently were thermally desorbed from the Tenax trap at 225°C for 4 min with a helium flow rate of 300 mL min^{-1}. The desorbed compounds were cryofocused at -30°C using liquid nitrogen at the entrance of a DB-624 capillary column (JandW scientific, Folsom, CA, USA).

Gas Chromatography/Mass Spectrometry (GC/MS): A gas chromatograph 6890N (Agilent Technologies Spain, S.L., Madrid, Spain) equipped with mass detector 5973N (Agilent Technologies Spain, S.L., Madrid, Spain) was used with a DB-624 capillary column (JandW scientific: 30 m×0.25 mm id, 1.4 μm film thickness). The sample was injected in split mode (1:20). Helium was used as a carrier gas with a linear velocity of 36 cm/s. The temperature program used was as follows: 40°C maintained for 2 min and then raised from 40 to 100°C at 3°C/min, then from 100 to 180°C at 5°C/min and from 180 to 250°C at 9°C/min with a final holding time of 5 min; total run time 50.8 min. Injector and detector temperatures were set at 220 and 260°C, respectively.

The mass spectra were obtained using a mass selective detector working in electronic impact at 70 eV, with a multiplier voltage of 1953 V and collecting data at a rate of 6.34 scans/s over the range m/z 40-300. Compounds were identified comparing their mass spectra with those contained in the NIST05 (National Institute of Standards and Technology, Gaithersburg) library and/or by calculation of retention index relative to a series of standard alkanes (C_5-C_{19}) (Supelco 44585-U, Bellefonte, PA, USA) and matching them with data reported in literature. Samples were analyzed in triplicate. Results were reported as relative abundance expressed as total area counts (AU×10^6).

Statistical analysis: For the statistical analysis of the results of instrumental texture measurements, free fatty acid, free amino acid, volatile compounds and physico-chemical traits an analysis of variance (ANOVA) of one way using IBM SPSS Statistics 19.0 (IBM Corporation, Somers, NY, USA) was performed for all variables considered in the study. The Least Squares Mean (LSM) were separated using Duncan's t-test. All statistical test of LSM were performed for a significance level ($p<0.05$). Correlations between variables were determined by correlation analyses using the Pearson´s linear correlation coefficient with above statistical software package mentioned.

RESULTS AND DISCUSSION

Table 2 shows the physico-chemical properties, colour parameters and instrumental texture measurements through the manufacture process of dry-cured Celta "lacón". Significant differences ($p<0.001$) during the whole of the manufacturing process were observed on pH values. An increase on final pH values in comparison with pH value of raw material (from 5.64 to 6.04) was found. Our final pH values were slightly lower than those reported by other authors (Marra *et al*., 1999; Lorenzo *et al*., 2003; Lorenzo *et al*., 2008b; Veiga *et al*., 2003) in dry-cured "lacón" and it is the same order those reported by Vestegaard *et al*. (2000) for dry-cured hams (5.5-6.2). According to these results, Celta "lacón" does not undergo true lactic fermentation and are in agreement with those found previously by others authors (Marra *et al*., 1999; Lorenzo *et al*., 2003). The increase on pH values through the manufacture process could be related with low-weight nitrogen molecules and ammonia formation ascribed to endogenous and exogenous proteolytic enzyme activities (Lucke, 1994; Virgili *et al*., 2007).

The mean moisture content observed in raw pieces (74.82%) is similar to the values found by other authors (Veiga *et al*., 2003). This content decreases progressively during the whole of the curing process being more pronounced during the last step. Our average final values (58.7%) were similar to observed by others authors (Veiga *et al*., 2003) in "lacón" samples with the same dry-ripening time than ours, while Lorenzo *et al*. (2008a) found

Table 2: Evolution of physico-chemical parameters during the manufacture of dry-cured "lacón", results expressed as means±standard error (n=5)

	Fresh piece	Salting	Post-salting	Dry-ripening	SEM	SIG
Chemical composition						
pH	5.64±0.09^{a}	5.58±0.08^{a}	5.63±0.05^{a}	6.04±0.10^{b}	0.05	***
Water activity	0.989±0.001^{c}	0.937±0.016^{b}	0.926±0.009^{b}	0.908±0.006^{a}	0.01	***
Moisture (%)	74.82±0.59^{d}	70.86±1.70^{c}	67.49±0.36^{b}	58.67±0.99^{a}	1.38	***
Intramuscular fat (%)	3.64±1.32	4.76±0.30	4.41±1.39	5.22±1.44	0.64	n.s.
Protein (%)	82.95±1.23^{d}	76.88±2.17^{c}	72.04±2.03^{b}	68.61±2.16^{a}	1.30	***
TBAR´S (mg MDA kg^{-1})	0.18±0.03^{a}	0.98±0.43^{b}	3.50±0.51^{d}	2.60±0.61^{c}	0.31	***
NaCl (% T.S.)	0.85±0.19^{a}	10.76±0.36^{b}	14.95±0.47^{c}	15.63±0.50^{d}	1.35	***
WHC						
Cooking Loss (%)	22.66±3.19^{d}	18.05±2.60^{c}	13.92±1.51^{b}	4.37±1.38^{a}	1.61	***
Colour parameters						
Luminosity (L*)	38.68±0.70^{c}	33.22±2.07^{ab}	31.82±1.27^{b}	35.13±2.39^{b}	0.68	***
Redness (a*)	19.10±0.64^{b}	18.70±2.48^{b}	14.18±1.43^{a}	14.55±2.29^{a}	0.64	**
Yellowness (b*)	10.05±0.54^{b}	10.25±1.36^{b}	8.58±0.36^{a}	7.67±1.03^{a}	0.30	**
Textural Parameters						
Shear force (kg cm^{-2})	3.80±0.66^{a}	4.72±0.85^{a}	7.30±0.70^{b}	11.73±1.01^{c}	0.72	***
Firmness (kg cm^{-2})	0.95±0.18^{a}	1.43±0.23^{b}	1.23±0.31^{ab}	2.59±0.45^{c}	0.16	***
Total Work (kg*s)	16.55±1.40^{a}	18.73±1.34^{a}	23.56±1.78^{b}	55.23±2.89^{c}	3.60	***
TPA test						
Hardness (kg)	6.78±0.46^{a}	7.97±0.87^{a}	11.69±1.27^{b}	20.66±0.85^{c}	1.26	***
Springiness (mm)	0.51±0.05	0.50±0.04	0.52±0.03	0.55±0.03	0.01	n.s.
Chewiness (kg* mm^{-1})	2.10±0.31^{a}	2.48±0.45^{a}	3.62±0.39^{b}	7.03±0.18^{c}	0.45	***
Gumminess (kg)	4.84±0.96^{a}	4.56±0.75^{a}	6.87±0.47^{b}	11.86±0.43^{c}	0.68	***
Cohesiveness	0.58±0.05^{ab}	0.59±0.03^{b}	0.56±0.01^{ab}	0.54±0.03^{a}	0.01	n.s.

$^{a-d}$Means in the same row with different letters differ significantly ($p<0.05$, Duncan test) significance levels: *$p<0.05$, **$p<0.01$, ***$p<0.001$, n.s.: not significant

final values below 50%. Pearson correlation test indicated that moisture contents were positively related to instrumental colour attributes of a* values (r = 0.60, $p<0.01$) and b* values (r = 0.68, $p<0.01$) and negatively correlated to pH (r = -0.83, $p<0.01$). In the same line, water activity gradually declined over the curing period from an initial value of 0.989 to 0.901 ($p<0.001$), on average (Table 2). This decrease, is due to the decrease of the water content, salt diffusion and the intense dehydration that the pieces undergo during the drying-ripening stage, in fact a_w values showed a positive correlation with moisture content (r = 0.85, $p<0.01$) and a negative correlation with salt content (r = -0.95, $p<0.01$). The Pearson correlations indicated that a_w was also related to the hardness (r = -0.74), gumminess (r = -0.67), chewiness (r = -0.71), L* values (r = 0.56), a* values (r = 0.62) and b* values (r = 0.59).

On the other hand, salt content (expressed as g/100 g of Total Solids) showed significantly ($p<0.001$) increased during the salting and post-salting stages as result of salt diffusion throughout the whole piece, reaching values that remained relatively constant until the end of the manufacture process (Table 2). The final mean values (15.63%) were higher than those reported by others authors for "lacón" (Marra *et al*., 1999; Lorenzo *et al*., 2008a; Veiga *et al*., 2003) who found values ranged 13 and 18.67 % of TS. However, Garrido *et al*. (2012) showed final average NaCl contents of 11.23, 12.22 and 12.75 g NaCl/100 g of TS in dry-cured "lacón" salted for 3, 4 and 5 days, respectively. Our sodium chloride values are found above of the range (5-9%) of those observed by other authors in other dry-cured meat products (Guerrero *et al*., 1999; Melgar *et al*., 1990). Intramuscular fat did not show significant differences ($p>0.05$) along the ripening process. Our mean values (4.50%) were lesser than those reported by other authors for dry cured "lacón" (Marra *et al*., 1999; Lorenzo *et al*., 2003). Several studies have showed different levels of intramuscular fat for Iberian ham (Petron *et al*., 2004; Ventanas *et al*., 2007), Serrano ham (Gandemer, 2009; Jimenez-Colmenero *et al*., 2010), Bayonne ham (Gandemer, 2009) and Parma ham (Lo Fiego *et al*., 2005). Gandemer (2009) and Ruiz-Carrascal *et al*. (2000) noticed that intramuscular fat content is the parameter that more affects the appearance, texture and flavor of dry-cured hams. A decrease in the protein content was observed during the manufacturing process, from an initial average value of 82.95 to 68.61% of TS at the end of ripening stage. This decrease appears to be fundamentally due to the increase in the NaCl content and decrease of moisture during the manufacture process.

A similar trend as in moisture profile was found in the water-holding capacity measured by Cooking Loss (CL). During the dry-ripening stage the CL was dramatically affected ($p< 0.001$) decreasing the value from 22.6 to 4.4%. The Pearson correlations indicated that CL was positively related to the a* values (r = 0.59, $p<0.01$), b* values (r = 0.65, $p<0.01$), aw (r = 0.80, $p<0.01$), moisture content (r = 0.96, $p<0.01$) and protein content (r = 84, $p<0.01$) and negatively related to the pH (r = -0.77, $p<0.01$), ClNa

content (r = -0.79, p<0.01), hardness (r = -0.93, p<0.01), gumminess (r = -0.87, p<0.01) and chewiness (r = -0.91, p<0.01). The denaturation of meat protein caused decreases of the WHC (Gratacos-Cubarsi and Lametsch, 2008) and this led to water loss during the processing of dry-cured of "lacón". Also, Alvarado and McKee (2007) noted that the irreversible reduction of protein functionality in meat related to the alteration of WHC may affect the rate of water lost from the meat including free, immobilized and bound water.

The degree of oxidation in the dry-cured "lacón" was measured by TBAR´S method which evaluates malonaldehyde formed in oxidation process. TBAR´S values increased significantly (p<0.001) during the salting stage and during the post-salting period from 0.18 to 0.98, 3.50 mg malonaldehyde/kg of muscular portion, respectively. The increase in malonaldehyde contents during the salting and post-salting stages was also reported by Garrido *et al.* (2009) in "lacón" samples and by Melgar *et al.* (1990) in hams. From these maximum values, a significant (p<0.001) drop was observed at the end of process reaching final average values of 2.60 mg MDA kg^{-1} of muscular portion. Our final values were the same order of those reported by other authors (Garrido *et al.*, 2009; Veiga *et al.*, 2003) in "lacón" samples and slightly lower than those observed by Rodriguez *et al.* (2001) and by Lorenzo *et al.* (2008b). This final drop was attributed to the instability of the malonaldehyde (Melgar *et al.*, 1990).

The influence of ripening time on colorimetric characteristics is shown in Table 2. The luminosity (L*) values decreased during salting and post-salting stage from 38.7 to 33.2 and 31.8, respectively. These results are in agreement with those found by other authors (Marusic *et al.*, 2011; Perez-Palacios *et al.*, 2011) for dry-cured ham. This decrease was significantly correlated with a_w (r = 0.55, p<0.05), protein content (r = 0.50, p<0.05) and ClNa content (r = -0.71, p<0.01). On the other hand, a* values showed a decrease during the post-salting stage and then remained constant until the end of process. This final value was similar to that described by Cava *et al.* (2003) for dry-cured loin and higher than those reported by Perez-Alvarez *et al.* (1999) Andres *et al.* (2000) for dry cured ham. Finally, yellowness values decreased as the time in process increased and this decrease was more pronounced during post-salting stage (p< 0.001). A similar pattern has been reported by Andres *et al.* (2000) for Iberian ham. These differences observed between dry-cured "lacón" and dry-cured ham could be due to the processing time, which is much shorter in the case of dry cured "lacón" with respect to the dry cured ham.

The parameters obtained from the Warner Bratzler (WB) shear test are presented in Table 2. Shear force showed a marked rise (p<0.001) from raw piece (3.80 kg/cm^2) to the end of process (11.73 kg cm^{-2}). This final value was higher than those reported by Guerrero *et al.* (1999) for dry-cured ham and by Ramirez and Cava (2007) for dry-cured loin. On the other hand, total work followed a similar trend showed the maximum values at the end of process of 55.23 kg*s. The texture profile of dry-cured Celta "lacón" (hardness, springiness, chewiness, gumminess and cohesiveness) was followed during the ripening period and results are given in Table 2. Hardness, chewiness and gumminess were affected by processing time (p<0.001) whereas springiness and cohesiveness did not show significant differences (p>0.05). Hardness increased (p<0.001) from 6.78 to 20.66 kg during ripening. Changes in hardness during dry-cured meat products ripening have been attributed to both water content and state of protein (Monin *et al.*, 1997). It was found from Pearson correlation test that hardness was related (p<0.01) to pH, moisture content, a_w, cooking loss and protein content with correlation coefficients of r = 0.85, r = -0.96, r = -0.74, r = -0.93 and r = 0.82, respectively. Moisture content, cooking loss and a_w were negatively correlated to hardness so that decreasing moisture content, cooking loss and a_w increased (p<0.01) hardness. Our results of hardness were higher than those reported by Guerrero *et al.* (1999) for dry-cured hams due that samples used for TPA analysis were cooked before analysis. On heat treatments at ambient pressure it is seen that there is an increase in hardness probably attributed to changes in the myofibrillar components of the muscle. In dry cured ham, texture is a characteristic directly related to the muscle structure, especially related to the degradation of myofibrillar protein and collagen as well as to the intramuscular fat content and drying rate (Toldra and Flores, 1998). Gumminess and chewiness values increased (p<0.001) from 4.84 to 11.86 kg and from 2.10 to 7.03 kg*mm during ripening period, respectively (Table 2), while cohesiveness and springiness did not show significant differences during the process. It was found from the Pearson test that moisture content and a_w were related (p<0.01) to chewiness (r = -0.95 and r = -0.71, respectively) and gumminess (r = -0.92 and r = -0.67, respectively) values.

Changes of free fatty acid: The changes in the content of the different Free Fatty Acids (FFA) during the manufacture process of dry-cured Celta "lacón" are shown in Table 3. The total average content of FFA increased significantly (p<0.05), from 24.62 mg g^{-1} of fat in the raw pieces to 54.41 mg g^{-1} of fat at the end of the drying-ripening stage. It was reported that the amount of

Table 3: Evolution of free fatty acids during the manufacture of dry-cured "lacón", results expressed as means±standard error (n = 5)

Fatty acids	Fresh piece	Salting	Post-salting	Dry-ripening	SEM	SIG
C16:0	7.11±1.41^{a}	7.27±0.43^{a}	10.24±0.74^{b}	14.67±1.15^{c}	0.73	***
C18:0	2.83±0.54^{a}	3.50±0.29^{b}	5.94±0.52^{c}	6.17±0.50^{c}	0.35	***
C18:1*cis*-9	7.42±1.07^{a}	9.18±0.70^{b}	15.50±0.87^{c}	19.13±1.74^{d}	1.11	***
C18:2*n*-6	6.26±0.72^{a}	8.23±1.44^{b}	7.78±1.35^{b}	12.23±0.60^{c}	0.55	***
C20:4*n*-6	0.99±0.16^{a}	1.28±0.30^{a}	2.00±0.44^{b}	2.19±0.63^{b}	0.14	**
SFA	9.95±1.80^{a}	10.78±0.58^{a}	16.19±1.09^{b}	20.85±1.14^{c}	1.04	***
MUFA	7.42±1.07^{a}	9.18±0.70^{b}	15.50±0.87^{c}	19.13±1.74^{d}	1.11	***
PUFA	7.25±0.83^{a}	9.72±1.91^{a}	9.53±2.11^{a}	13.54±2.78^{b}	0.66	**
Total FFA	24.62±3.24^{a}	29.49±1.89^{b}	41.48±1.95^{c}	54.41±3.53^{d}	2.70	***

Results expressed as mg g^{-1} of fat, $^{a-d}$Means in the same row with different letters differ significantly ($p<0.05$, Duncan test) Significance levels: *$p<0.05$, **$p<0.01$, ***$p<0.001$, n.s.: not significant PUFA = $\sum$ (C18:2n-6+C20:4n-6), MUFA =$\sum$ (C18:1*cis*-9), SFA = $\sum$ (C16:0+C18:0)

Table 4: Evolution of free amino acids during the manufacture of dry-cured "lacón", results expressed as means±standard error (n=5)

Amino acids	Fresh piece	Salting	Post-salting	Dry-ripening	SEM	SIG
Histidine	73.87±9.24^{a}	80.41±7.26^{b}	123.66±4.04^{c}	135.83±16.20^{c}	4.42	***
Isoleucine	15.73±1.82^{a}	16.79±2.70^{a}	24.34±1.16^{a}	101.89±11.80^{b}	8.37	***
Leucine	26.37±1.23^{a}	28.47±3.93^{a}	48.04±2.11^{b}	178.68±20.88^{c}	14.63	***
Lysine	25.24±0.36^{a}	27.59±3.45^{a}	50.95±3.73^{b}	247.24±22.58^{c}	21.38	***
Methionine	7.76±1.28^{a}	8.18±1.11^{a}	16.23±2.03^{b}	60.07±3.82^{c}	4.98	***
Phenylalanine	16.13±1.82^{a}	16.03±1.66^{a}	23.74±2.17^{a}	86.48±12.11^{b}	6.89	***
Threonine	56.70±6.74^{a}	61.33±10.48^{a}	79.39±12.54^{b}	156.96±7.73^{c}	9.47	***
Taurine	95.02±7.32^{a}	101.84±4.54^{b}	303.96±12.66^{c}	411.37±62.05^{d}	31.67	***
Valine	21.70±1.34^{a}	23.65±4.83^{a}	34.66±1.42^{b}	147.54±13.39^{c}	12.15	***
Arginine	119.13±7.67^{a}	134.20±6.24^{a}	454.90±31.05^{b}	663.45±81.37^{c}	53.19	***
Alanine	73.08±8.46^{a}	83.57±8.66^{a}	174.71±10.50^{b}	278.77±11.50^{c}	19.12	***
Aspartic acid	7.68±3.04^{a}	10.10±2.30^{a}	7.95±2.47^{a}	88.39±9.28^{b}	8.00	***
Cysteine	5.29±0.76^{a}	6.26±1.04^{a}	9.28±1.31^{b}	15.66±2.89^{c}	0.99	***
Glutamic acid	39.79±5.32^{a}	46.27±2.69^{a}	68.44±7.17^{b}	279.65±27.54^{c}	22.98	***
Glycine	42.74±1.49^{a}	38.42±2.98^{a}	41.78±6.66^{a}	95.99±7.20^{b}	5.57	***
Proline	11.07±0.57^{a}	13.99±2.72^{ab}	22.81±5.20^{b}	130.61±13.18^{c}	11.52	***
Serine	34.68±2.28^{ab}	30.56±2.34^{a}	45.07±6.65^{b}	146.67±15.88^{c}	11.13	***
Tyrosine	14.87±1.73^{a}	17.01±2.52^{a}	28.77±2.19^{b}	80.55±15.93^{c}	6.34	***
Hydroxiproline	1.65±0.31^{a}	1.54±0.23^{a}	1.81±0.47^{a}	4.06±0.15^{b}	0.24	***
Total	688.58±5.19^{a}	738.26±40.55^{a}	1562.93±56.94^{b}	3309.94±658.49^{b}	245.25	***

Results expressed as mg/100 g on the basis of dry matter, $^{a-d}$Means in the same row with different letters differ significantly ($p<0.05$, Duncan test), Significance levels: *$p<0.05$, **$p<0.01$, ***$p<0.001$, n.s.: not significant

FFA increased in other meat products, during the processing, such as: dry-cured hams (Zhou and Zhao, 2007), dry-cured sausages (Navarro *et al*., 1997) and dry-cured loins (Hernandez *et al*., 1999). As the muscle enzyme systems play an important role in the generation of FFA (Motilva *et al*., 1992), the increase in the amounts of free fatty acids could be the result of the action of lipolytic enzymes.

The main FFA in the raw pieces were oleic acid ($C_{18:1cis9}$), followed by linoleic ($C_{18:2n-6}$) and palmitic ($C_{16:0}$). This FFA profile is consistent with that reported by Veiga *et al*. (2003), Lorenzo *et al*. (2008b) for raw pieces of "lacón" and also with those showed by different authors for raw hams (Martin *et al*., 1999; Timon *et al*., 2002). As ripening time increase, an increase in oleic acid (from 7.14 to 19.13 mg g^{-1} of fat; $p<0.001$) and palmitic acid (from 7.11 to 14.67 mg g^{-1} of fat; $p<0.001$) was found. The greatest increase in levels of these FFA took place during the drying-ripening stage, which is consistent with the findings of Lorenzo *et al*. (2008b). A similar trend as in oleic acid was found in linoleic ($C_{18:2n-6}$) and arachidonic ($C_{20:4n-6}$) acids, which significantly increase ($p<0.001$) during the manufacture of dry-cured "lacón". Increases in linoleic and arachidonic acid contents have also been described in other dry-cured meat products (Lorenzo *et al*., 2008b), although some authors (Martin *et al*., 1999) have observed a decrease in the contents of these acids.

The final amount of each individual fatty acid should be the result of the balance between its release from glycerides and phospholipids and its oxidative degradation. The main FFA at the end of the manufacturing process was oleic ($C_{18:1cis9}$), followed by linoleic ($C_{18:2n-6}$), palmitic acid ($C_{16:0}$) and stearic ($C_{18:0}$) (Table 3).This FFA profile is similar to that described by other authors for ham (Coutron-Gambotti *et al*., 1999; Martin *et al*., 1999) and dry-cured "lacón" (Lorenzo *et al*., 2008b).

Changes of free amino acid: Table 4 shows the evolution of Free Amino Acids (FAA) content during the manufacture of dry-cured "lacón". These free amino acids have been usually identified when studying dry-cured ham (Ruiz *et al*., 1999; Toldra *et al*., 2000). Ornithine,

which has a non-protein origin, has been previously reported in dry-cured ham (Toldra *et al*., 2000) but it was not identified in this study, which matches previous results in Iberian ham (Cordoba *et al*., 1994; Martins *et al*., 2001; Ruiz *et al*., 1999). Cysteine, a free amino acid coming from proteolysis, was detected, which disagrees with previous studies on dry-cured ham (Ruiz *et al*., 1999; Toldra *et al*., 2000).

A significant increase ($p<0.001$) on the total free amino acids was observed from raw pieces (688.6 mg/100 g of TS) to the end of dry-ripening (3309.9 mg/100 g of TS). These final contents were higher than those reported in previous studies (Lorenzo *et al*., 2008b; Garrido *et al*., 2012), who observed final values below 3000 mg/100 g of TS in dry-cured "lacón". However, these final contents were lower than those reported by other authors (Monin *et al*., 1997; Ruiz *et al*., 1999; Virgili *et al*., 2007) for cured ham.

The increase in the individual free amino acids observed during the process was consistent with the increase in the total free amino acid content. This increase differed in the different amino acids: histidine, glycine, hydroxiproline, threonine and cysteine suffered the least increase; leucine, valine, isoleucine, tyrosine, phenylalamine, arginine, taurine, serine and alamine underwent moderate increases; and proline, aspartic acid, lysine, methionine and glutamic acid underwent the greatest increases. Cordoba *et al*. (1994) observed that in Iberian hams the free amino acids that underwent the greatest increases during the whole manufacture process were alamine and glutamic acid, followed by leucine, glycine and lysine, which is similar to the present findings. Schivazappa *et al*. (1995) reported that the quantity and type of free amino acids formed during manufacturing of ham depended on the activity of the aminopeptidases, cathepsins and muscle peptidases and of the NaCl content and the water activity values, variables that influence these enzymatic activities.

Arginine and taurine were the most abundant free amino acid found in raw pieces and are in disagreement with reported by Lorenzo *et al*. (2008b) who showed that the main free amino acid in the fresh pieces was glutamine, followed by taurine and alamine. The aminopeptidase activity is considered the main process implied in the FAA release in meat. However, the enzymatic activity is negatively affected by the relative increasing concentration of the salt occurring during drying and ripening due to water evaporation and, as a consequence, by the correspondent changes in the physico-chemical properties of the matrix (i.e., aw) (Garcia-Garrido *et al*., 2000).

The most abundant free amino acids detected in the final product were arginine, followed by taurine, glutamic acid and alanine, which were up to 270 mg/100 g of TS. This free amino acid profile differ with those observed in a previous study (Lorenzo *et al*., 2008b) where lysine, glutamic acid, γ-aminobutyric, glutamine and serien were the most abundant free amino acids at the end of the manufacturing process of dry-cured "lacón".

Free amino acids constitute a potential source of volatile compounds as follows: (i) through Strecker degradation of valine (Val), isoleucine (Ile) and leucine (Leu), giving 2-methyl-propanal, 2-methyl-butanal and 3-methylbutanal, (ii) generation of dimethyl disulfide compounds from sulfur-containing amino acids, such as methionine, cysteine and cystine and (iii) the generation of a several pyrazines from Maillard reactions (Toldra *et al*., 2000).

Changes of volatile compounds: Sixty four volatile compounds were identified during the manufacture of dry-cured Celta "lacón" (Table 5) and classified into 10 chemical families (aldehydes, aromatic hydrocarbons, alcohols, esters, aliphatic hydrocarbons, furans, ketones, branched hydrocarbons, acids and other compounds). An increase in the total amount of volatile compounds was observed during the whole process, from an initial average value of 140.03×10^6 to 1932.70×10^6 area units ($p<0.001$) (Fig. 1). At the end of drying-ripening process the volatile compounds profile maintained the relationship esters > aliphatic hydrocarbons > branched hydrocarbons > alcohols > ketones > aldehydes > other compounds > aromatic hydrocarbons > furans > acids. These results are in agreement whit those reported by other authors (Barbieri *et al*., 1992; Bolzoni *et al*., 1996) who confirmed the esters as the family of compounds of highest percentage among the volatile components of Parma ham. In opposite, in other dry-cured hams (Gaspardo *et al*., 2008; Zhang *et al*., 2006) esters were found at low percentages. However, other authors (Huan *et al*., 2005; Purrinos *et al*., 2011a; Sanchez-Pena *et al*., 2005) reported aldehydes like the most abundant chemical family in different dry-cured meat products, while Gaspardo *et al*., 2008 concluded that alcohols family were the most abundant in "San Daniele" ham.

Esters showed a marked increase ($p<0.001$) during the curing process of dry-cured "lacón", representing about 34.52% at the end of dry-ripening stage (Fig. 1). These compounds are formed by the esterification of carboxylic acids and alcohols. The enzymes involved in this reaction are esterase enzymes which are produced by different microorganisms (yeasts, moulds and bacteria)

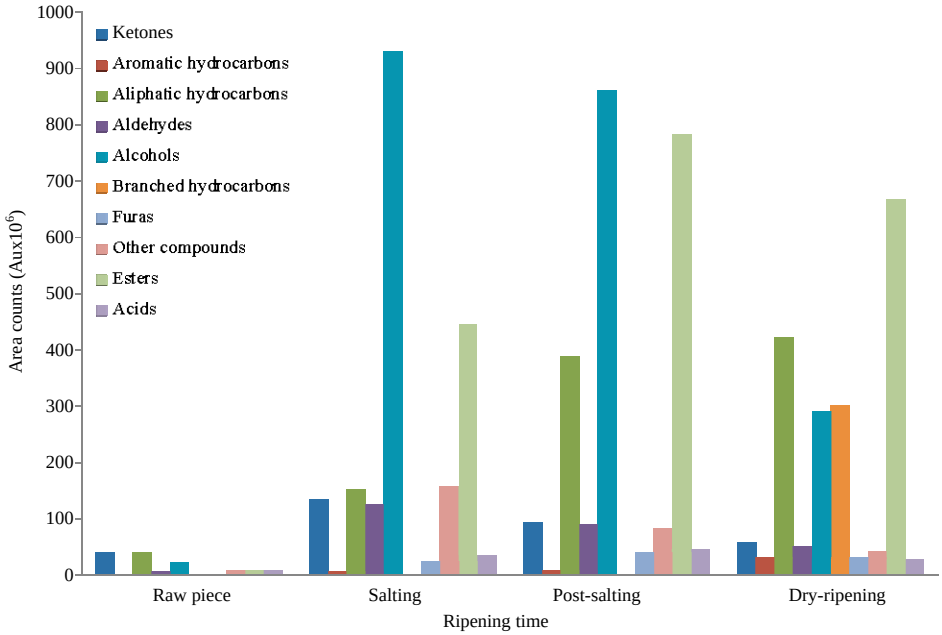

Fig. 1: Evolution of volatile compounds during the manufacture of dry-cured "lacón"

(Stahnke *et al*., 2002). They can modulate the global flavour due to their low odour thresholds, imparting fruity notes (Marco *et al*., 2007). Among these compounds, the most abundant at the end of process was hexanoic acid, methyl ester, reaching values of 267.1×10^6 area units, followed by acetic acid, methyl ester and butnaoic acid, methyl ester, which represented around 83% of the total esters. Esters strongly affect the flavour of ham as typical aged meat products; in particular, the methyl branched short-chain esters were found to be positively related to the attribute of aged meat (Careri *et al*., 1993). The detection of ethyl esters, such as acetic acid, methyl ester and propanoic acid, methyl ester, could be explained by the esterification activity of staphylococci and lactic acid bacteria (Talon *et al*., 1998).

Aliphatic hydrocarbons increased significantly ($p<0.001$) from 40 to 423×10^6 area units during the whole process (Fig. 1). Significant differences were obtained for all aliphatic hydrocarbons detected during curing process (Table 5). Some authors (Ansorena *et al*., 2001; Ruiz *et al*., 2002) observed that aliphatic hydrocarbons with less than ten carbons atoms as pentane, hexane, heptane and octane were the most abundant at the end of process, which derive from lipid oxidation (Muriel *et al*., 2004), while those with longer chains could be accumulated in the fat depots of the animal, probably from feeding (Meynier *et al*., 1998). It was remarkable the high total value of aliphatic hydrocarbons at the end of the drying-ripening stage (21.89% of total chromatographic area). However, these results are not relevant due to hydrocarbons are not important contributors to the meat product flavour because of their high odour thresholds (Bianchi *et al*., 2007).

On the other hand, branched hydrocarbons were also detected at the end of process, representing about 15.57% of total chromatographic area (Fig. 1). Among these family, the most abundant was hexane, 2, 2,3- trimethyl, reaching values of 119.7×10^6 area units, followed by nonane, 3,7-dimethyl and nonane, 5-methyl, which represented around 69.4% of the total branched hydrocarbons. Regarding aromatic hydrocarbons, only two volatile compounds from this family were detected during the whole process (Table 5). Among these compounds, p-xylene was the most abundant, showed the highest amount at the end of process (30.56×10^6 area units). Animal feeding, particularly grass consumption (Ruiz *et al*., 1999) and the smoked process (Ansorena *et al*., 2001) have been reported as the most probable origins for aromatic hydrocarbons in dry-cured ham and dry-fermented sausages, respectively.

The most abundant ketones detected at the end of dry-ripening process was 2-pentanone following by 2-heptanone (38.31 and 21.34×10^6 area units, respectively). On the other hand, 2,3-butanedione and 2-butanone only appeared in the first stages of processing. 2-ketones have been abundantly isolated in dry cured products

Table 5: Evolution of volatile compounds (chromatographic peak area) during the manufacture of dry-cured "lacón", results expressed as means±standard error (n=5)

Chemical class (AU×10^6)	LRI	K	Fresh piece	Salting	Post-salting	Dry-ripening	SEM	Sig
Ester compounds								
Acetic acid, methyl ester	559	m, k	0.00±0.00^a	199.51±46.20^b	341.54±44.89^c	164.45±34.65^b	28.87	***
Propanoic acid, methyl ester	689	m, k	0.00±0.00^a	0.00±0.00^a	10.79±0.66^b	25.37±3.33^c	2.41	***
Butanoic acid, methyl ester	802	m, k	0.00±0.00^a	66.22±11.39^b	97.81±8.29^c	119.97±18.67^c	10.65	***
Pentanoic acid, methyl ester	864	m, k	0.00±0.00^a	10.17±1.97^b	19.45±3.66^c	24.62±0.66^d	2.20	***
Hexanoic acid, methyl ester	995	m, k	0.00±0.00^a	83.20±10.56^b	225.95±50.95^c	267.10±48.88^c	25.75	***
Acetic acid, hexil ester	1088	m, k	0.00±0.00^a	0.00±0.00^a	6.40±1.19^b	0.00±0.00^a	0.65	***
Heptanoic acid, methyl ester	1100	m	0.00±0.00^a	5.85±0.76^b	12.91±1.99^c	0.00±0.00^a	1.24	***
Octanoic acid methyl ester	1188	m, k	0.00±0.00^a	7.22±0.80^b	12.00±1.21^c	17.48±2.03^d	1.50	***
Nonanoic acid methyl ester	1266	m, k	0.00±0.00^a	0.00±0.00^a	10.59±1.88^c	5.10±0.36^b	1.02	***
Ethylacetate	626	m, k	9.81±5.56^b	57.32±4.03^d	29.56±1.72^c	0.00±0.00^a	5.08	***
Methyl propionate	643	m, k	0.00±0.00^a	7.63±0.76^b	7.89±0.79^b	7.67±0.64^b	0.78	***
3-Hydroximandelic acid methyl ester d-TMS	1182	m, k	0.00±0.00^a	8.95±1.05^b	7.67±0.55^b	35.69±5.52^c	3.15	***
Aliphatic hydrocarbons								
Pentane	500	m, k	0.00±0.00^a	5.42±0.97^b	24.35±2.47^c	25.63±5.78^c	2.68	***
Hexane	600	m, k	27.87±3.30^a	69.75±3.41^b	89.79±2.97^c	64.48±8.13^b	20.69	***
Heptane	700	m, k	1.44±0.37^a	38.27±7.81^b	138.85±12.59^c	163.81±12.73^d	15.64	***
Octane	800	m, k	10.87±2.44^a	20.50±5.83^b	109.88±4.69^c	160.90±18.54^d	14.51	***
Decane	1000	m	0.00±0.00^a	2.09±0.39^b	3.56±0.98^c	4.99±0.62^d	0.44	***
Undecane	1100	m, t	0.00±0.00^a	1.79±0.40^b	4.21±0.74^c	0.00±0.00^a	0.41	***
Dodecane	1200	m, t	0.00±0.00^a	4.48±0.30^b	5.30±0.75^c	0.00±0.00^a	0.50	***
Tridecane	1300	m, t	0.00±0.00^a	2.63±0.34^c	2.82±0.81^c	1.40±0.06^b	0.27	***
Tetradecane	1400	m, t	0.00±0.00^a	2.11±0.13^c	3.59±0.31^d	0.92±0.17^b	0.31	***
Pentadecane	1500	m	0.00±0.00^a	0.00±0.00^a	1.11±0.15^b	0.00±0.00^a	0.11	***
2,4-octadiene	889	m, t	0.00±0.00^a	5.96±0.49^b	5.77±0.69^b	0.00±0.00^a	0.68	***
Branched hydrocarbons								
Hexane, 3-ethyl	874	m, t	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	1.85±0.53^b	0.19	
Hexane, 2,2,3-trimethyl	1120	m, t	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	119.67±10.35^b	11.94	***
Hexane, 2,2,5,5-tetramethyl-	902	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	20.98±5.10^b	2.15	***
Heptane, 3-ethyl-	892	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	2.66±0.64^b	0.27	***
Heptane, 2, 4, 6-trimethyl-	897	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	1.92±0.11^b	0.19	***
Heptane, 2,2,3,5-tetramethyl-	912	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	14.35±2.71^b	1.45	***
Octane, 3,3-dimethyl-	954	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	25.48±4.72^b	2.58	***
Octane, 2-methyl-	883	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	0.97±0.15^b	0.10	***
Nonane, 5-methyl-	1098	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	43.25±5.98^b	4.34	***
Nonane, 5-methyl-5-propyl-	1103	m	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	24.31±6.36^b	2.50	***
Nonane, 3,7-dimethyl-	883	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	45.87±5.64^b	4.59	***
Aromatic hydrocarbons								
Benzene	661	m	0.00±0.00	6.97±0.43^c	7.93±0.73^c	1.93±0.08^b	0.77	***
P-xylene	921	m	3.23±0.18^b	0.00±0.00^a	0.00±0.00^a	30.56±11.38^c	3.18	***
Ketones								
2,3-butanedione	615	m, k	23.25±8.58^b	60.78±6.16^c	0.00±0.00^a	0.00±0.00^a	5.80	***
2-butanone	619	m, k	14.46±4.45^b	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	1.51	***
Methyl vinyl ketone	615	m, k	0.00±0.00^a	0.00±0.00^a	38.57±6.14^b	0.00±0.00^a	3.88	***
2-pentanone	711	m, k	1.30±0.20^b	29.84±1.91^c	0.00±0.00^a	38.31±10.68^d	4.05	***
2-heptanone	974	m, k	0.71±0.72^a	41.39±6.09^c	46.15±25.39^c	21.34±2.73^b	4.93	***
2-octanone	1083	m, k	0.00±0.00^a	4.15±0.95^b	8.70±0.80^c	0.00±0.00^a	0.83	***
Aldehydes								
Butanal, 3-methyl (3-methylbutanal)	671	m, k	4.59±1.89^a	56.59 ±5.02^c	67.21±2.11^d	36.35±6.83^b	5.55	***
Hexanal	850	m, k	1.78±0.63^a	14.27±4.33^{bc}	17.85±1.31^c	13.11±3.50^b	13.90	***
Heptanal	981	m, k	0.00±0.00^a	20.14±6.62^b	0.00±0.00^a	0.00±0.00^a	2.11	***
Octanal	1091	m, k	0.00±0.00^a	13.95±5.13^b	0.00±0.00^a	0.00±0.00^a	1.48	***
2-octenal	1156	m, k	0.00±0.00^a	0.00±0.00^a	2.72±0.35^b	0.00±0.00^a	0.27	***
2-dodecanal	1175	m, k	0.00±0.00^a	2.46±0.52^b	0.00±0.00^a	0.00±0.00^a	0.25	***
Nonanal	1183	m, k	0.00±0.00^a	18.59±6.10^d	3.79±0.30^c	2.69±0.14^b	1.78	***
Alcohols								
1-propanol	609	m, k	22.05±2.64^c	0.00±0.00^a	20.78±2.09^c	6.38±1.20^b	2.19	***
2-propanol	588	m, k	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	21.22±5.99^b	2.20	***
1-butanol	704	m, k	0.00±0.00^a	6.85±0.86^b	11.98±1.85^c	0.00±0.00^a	1.18	***
1-penten-3-ol	721	m, k	0.00±0.00^a	22.84±3.46^c	45.29±8.24^d	13.71±4.54^b	3.92	***
1-pentanol	821	m	0.00±0.00^a	353.21±50.21^c	334.87±17.51^c	156.20±31.23^b	33.67	***
1-hexanol	958	m, k	0.00±0.00^a	414.74±48.61^d	302.36±10.70^c	93.45±4.34^b	38.03	***
1-octen-3-ol	1074	m, k	0.00±0.00^a	132.43±19.69^b	145.39±45.03^b	0.00±0.00^a	16.75	***
Furans								
Furan, 2-ethyl	697	m, k	0.00±0.00^a	8.49±0.65^c	7.69±1.12^{bc}	6.46±0.55^b	0.78	***
2-n-butyl furan	940	m, k	0.00±0.00^a	1.51±0.12^b	5.29±0.79^c	0.00±0.00^a	0.50	***
Furan, 2-penthyl	1058	m, k	0.00±0.00^a	17.04±2.83^b	30.76±15.25^{bc}	25.58±4.25^c	3.14	***

Table 5: Continue

Chemical class (AU×10^6)	LRI	K	Fresh piece	Salting	Post-salting	Dry-ripening	SEM	Sig
Acids								
Acetic acid	684	m, k	8.43±3.23^a	36.46±6.31^c	46.48±4.57^d	29.42±2.20^b	3.32	***
Other compounds								
Metane,chloro	504	m, k	0.00±0.00^a	9.89±2.40^b	10.42±2.54^b	43.42±7.43^c	3.86	***
Hydrazine propil	570	m, k	0.00±0.00^a	69.25 ±6.94^b	0.00±0.00^a	0.00±0.00^a	6.92	***
Thiophene, 3-methyl	803	m, k	7.79±0.90^b	0.00±0.00^a	0.00±0.00^a	0.00±0.00^a	0.78	***
Camphene	1007	m, k	0.00±0.00^a	81.22±15.55^b	73.85±27.86^b	0.00±0.00^a	9.50	***

LIR: Linear retention indexes, calculated in relation to the retention time of n-alkane (C_5-C_{19}) series, R: Reliability of identification: k: Kovats index in agreement with literature (Flores *et al*., 1997; Marco *et al*., 2007; Olivares *et al*., 2010; Lorenzo *et al*., 2012); m: mass spectrum agreed with mass database (NIST05); t: tentative identification by mass spectrum, $^{a-d}$Means in the same row with different letters differ significantly ($p<0.05$, Duncan test), Significance levels: *$p<0.05$, **$p<0.01$, ***$p<0.001$, n.s. = not significant

(Muriel *et al*., 2004; Purrinos *et al*., 2011a) and they are considered to have a great influence on the aroma of meat and meat products.

Regarding aldehydes showed a marked increase from the raw piece to the end of salting step decreasing thereafter at the end of the drying-ripening stage ($p<0.001$). The following compounds were identified: 3-methylbutanal, hexanal, heptanal, octanal, 2-octenal, 2-dodecanal and nonanal. Typical oxidation products of n-3 and n-6 PUFAs are aldehydes such as pentanal, hexanal and heptanal, which may exhibit green, fatty and soapy aroma notes when present above their odour threshold concentrations. Pentanal and hexanal can be formed from decomposition of hydroperoxides during autoxidation of n-3 and n-6 fatty acids, respectively (Olsen *et al*., 2005). These compounds present a low olfaction threshold and are thus largely responsible for the final aroma produced in dry-cured ham (Muriel *et al*., 2004; Ramirez and Cava (2007)). The majority compounds at the end of process were 3-methylbutanal (36.35×10^6 area units) and hexanal (13.11×10^6 area units). On the other hand, nonanal is a main oxidation product of oleic acid (Belitz *et al*., 2001), which was the most abundant unsaturated fatty acid (Table 3), but in our study we did not find any correlation between both.

Only 1-propanol has been detected in raw pieces. During the manufactured process a total of 6 news alcohols were found (Table 5). At the end of dry-ripening stage 1-pentanol was the most abundant, representing about 54% of the total alcohols. Among unsaturated alcohols, 1-octen-3-ol showed high values in the salting and post-salting stages (Table 5). Jurado *et al*., 2009 reported than this compound could serve as an indicator for an acorn diet. It derives from oxidation of arachidonic acid which is important in pork meat (Wood *et al*., 2004). The total area of alcohols increased throughout salting, decreasing slightly during post-salting and afterwards followed decreasing until the end of drying-ripening process ($p<0.001$) (Fig. 1). Alcohols have a low odour threshold, so they are important contributors to the aroma of these products (Sabio *et al*., 1998).

With respect acids family, only acetic acid was detected in every sample point. Some authors have reported that this acid is originated by the fermentation of sugars by microorganisms (Kandler, 1983) and other by the Maillard reaction (Martins *et al*., 2001). However, its importance in dry-cured ham may be limited, because this compound was not described as an odour-active compound of ham (Carrapiso *et al*., 2002). Finally, furans were not detected in the raw material. The highest amount this family was observed at the end of the post-salting stage (Fig. 1). Among furans, 2-penthylfuran was the most abundant at the end of process, it is a noncarboxylic compound derived from linoleic acid and other n-6 fatty acids (Frankel, 1991), with relatively low threshold and vegetable aromatic note (Fay and Brevard, 2005). Numerous authors (Huan *et al*., 2005; Ruiz *et al*., 1999) have reported 2-pentylfuran among the headspace volatiles of a wide variety of dry-cured hams.

CONCLUSIONS

During manufacture process of "lacón" took place some reactions of proteolysis and lipolysis which affect the characteristics of final product. As ripening time increased in dry-cured "lacón", an increasing on TBAR´S values and hardness was observed. pH values showed a slight increase during the dry-ripening stage due to the formation of basic compounds probably as a result of proteolysis. FFA increased during the manufacture process, since oleic acid was the most abundant at the end of process followed by palmitic and linoleic.

In the final dry-cured "lacón", esters were the richest chemical family among flavour substances, followed by aliphatic hydrocarbons and branched hydrocarbons. The most abundant volatile compound was hexanoic acid, methyl ester. Flavour formation of dry-cured "lacón" began from salting stage.

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