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Changes in physico-chemical properties and volatile compounds throughout the manufacturing process of dry-cured foal loin

José M. Lorenzo [a,*], J. Carballo [b]

[a] Centro Tecnológico de la Carne de Galicia, Rúa Galicia N° 4, Parque Tecnológico de Galicia, San Cibrán das Viñas, 32900 Ourense, Spain
[b] Area de Tecnologia de los Alimentos, Facultad de Ciencias de Ourense, Universidad de Vigo, 32004 Ourense, Spain

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ABSTRACT

Physico-chemical, textural, lipolytic and volatile compound changes that occur during the manufacture of dry-cured foal loin were studied. Hardness and chewiness increased significantly ($P < 0.001$) from 1.67 kg and 0.48 kg * mm to 18.33 kg and 5.01 kg * mm, respectively during ripening process. The total average content of free fatty acid increased significantly ($P < 0.001$), from 768.8 mg/100 g of fat in the loins immediately after the seasoning period to 1271.1 mg/100 g of fat at the end of the drying-ripening period. In the final product, aldehydes became the dominant volatile compounds.

1. Introduction

Due to the continuous evolution of the economic and social conditions of industrialised countries food demands have been transformed and modified. Indeed, increasing consumer interest is being shown in the energetic and nutritional values of food, as well as in the role played by a correct diet and a healthy lifestyle. At present, consumers favour authentic meat, tasty, rich in protein and low in lipid and cholesterol content. Horse meat production in Spain reaches 7000 tonnes (FAOSTAT, 2012). Horse meat is characterized by low fat, low cholesterol contents (Lorenzo et al., 2014), and high levels of heme iron (Franco, Rodríguez, Purriños, Crecente, Bermúdez, & Lorenzo, 2011; Lorenzo, Pateiro, & Franco, 2013). From the point of view of fatty acid composition, horsemeat is characterized by high levels of unsaturated fatty acids (above 55%); polyunsaturated fatty acids (PUFA), predominantly the essential $n-6$ (linoleic acid, 18:2 $n-6$) and $n-3$ (linolenic acid, 18:3 $n-3$) PUFA and monounsaturated fatty acids (MUFA), primarily oleic acid (18:1 $n-9c$) (Lorenzo, 2013). These nutritional characteristics mean that this type of meat may be considered as a good alternative to beef meat.

Dry-cured meat products including dry-cured sausages, hams, dry-cured pork shoulder, and dry-cured beef and goat meats are one of the pillars of the Spanish food industry, their production reaching around 745,000 metric tonnes per year (MERCASA, 2013). The ripening process of dry-cured meat products involves complex chemical and biochemical changes in the main components of raw meat (proteins and lipids) leading to the generation of volatile compounds with distinct aromatic notes and/or olfaction thresholds (Ruiz, Muriel, & Ventanas, 2002). The acceptance of dry-cured products by consumers is mainly determinated by their sensory quality. The flavour is perhaps the most important quality parameter and it is markedly affected by raw material, processing techniques, and ageing time (Sánchez-Peña, Luna, García-Gomez, & Aparicio, 2005). Processing has a great influence on the final flavour of dry-cured loins, with both seasoning/curing ingredients addition and the ripening/drying phases involving complex chemical and biochemical changes in the main components of the raw meat (proteins and lipids) leading to the generation of volatile compounds, which are mainly esters and sulphide compounds (Lorenzo, 2014; Muriel, Antequera, Petrón, Andrés, & Ruiz, 2004). These compounds are responsible of the characteristic flavour of these products and they have influence on the consumer acceptance (Ruiz, García, Muriel, Andrés, & Ventanas, 2002).

Proteolysis and lipolysis are two of the most important processes that have an impact on final sensory quality. Proteolytic events have been shown to be an important source of aroma and flavour, as they release several compounds related to flavour development, such as free amino acids (Ordóñez, Hierro, Bruna, & de la Hoz, 1999). Lipolysis also plays an important role in the development of sensorial characteristics, because it causes an increase in free fatty acid content, and it also favours reactions such as oxidation, which leads to release of a large number of volatile compounds that are responsible for the characteristic flavour (Yang, Ma, Qiao, Song, & Du, 2005).

* Corresponding author. Tel.: +34 988 548 277; fax: +34 988 548 276.
E-mail address: jmlorenzo@ceteca.net (J.M. Lorenzo).

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Dry-cured pork loin is a meat product manufactured from the *longissimus thoracis et lumborum* muscle, broadly consumed and very appreciated in the entire world. There are some studies concerning the physico-chemical, microbial and sensorial characteristics of dry-cured pork loin (Muriel, Andrés, Petrón, Antequera, & Ruiz, 2007; Muriel et al., 2004; Ramírez & Cava, 2007; Ventanas, Estévez, Andrés, & Ruiz, 2008). However, as far as we know, dry-cured loin from foal is not produced in the world and there is not information about this meat product manufacture from foal meat. The aim of the present study was to investigate the changes on physico-chemical, textural, lipolysis and volatile compounds during the manufacture of dry-cured foal loin. This work aims, therefore, to explore the characteristics of a meat product that can be successful in the future due to the beneficial characteristics of foal meat already mentioned.

2. Materials and methods

2.1. Animals and manufacture of dry-cured foal loin

For this study, ten foals of the "Galician Mountain" breed were obtained from "Monte Cabalar" (agricultural cooperative of "Galician Mountain" breed) (A Estrada, Pontevedra, Spain). Foals were 15 months old, and carcasses weighed around 90.4 kg. A total of twenty loins with an average weight of 1.76 ± 0.24 kg, were selected. Loins were seasoned as whole pieces (53.2 cm length) following the usual way, by rubbing with 7.5 g per kg of supplement "Diana 655 AL" and 12 g per kg of supplement "Saboral Lomo Adobado B" from Cargill (Barcelona, Spain) composed, in unknown proportions, of sugar, salt, dextrin, olive oil, potassium nitrate, sodium nitrite, sodium ascorbate and spices such as Spanish paprika (*Capsicum annuum*, L.), oregano (*Origanum vulgare*, L.) and garlic (*Allium sativum*, L.). Loins were kept at 4 °C for 3 days to allow the seasoning mixture to penetrate. Then, the loins were stuffed into collagen casings and transferred to a post-seasoning room where they stayed for 30 days at 2–5 °C and 85–90% relative humidity. After the post-seasoning period, the pieces were transferred to a room at 12–14 °C and 74–78% relative humidity where a drying–ripening process took place for 60 days. The air convection in the drying room was intermittent and the air speed around the pieces when the fan was running ranged between 0.3 and 0.6 m/s. Samples were taken after the seasoning and post-seasoning periods and at 30 and 60 days of the drying ripening period. In each sample point, five pieces were randomly taken and analyzed.

2.2. Analytical methods

2.2.1. Reagents

Fatty acid methyl esters (FAMEs) 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).

2.2.2. pH, water activity, colour parameters and TBARS values

The pH of samples was measured using a digital pH-metre (Thermo Orion 710 A+, Cambridgeshire, UK) equipped with a penetration probe. Water activity was determinated using a Fast-lab (Gbx, Romans sur Isére Cédex, France) water activity metre, previously adjusted with sodium chloride and potassium sulphate. A portable colourimeter (Konica Minolta CM-600d, Osaka, Japan) with pulsed xenon arc lamp filtered to illuminant D65 lighting conditions, 0° viewing angle geometry and 8 mm aperture size, was used to estimate meat colour in the CIELAB space: lightness, (L*); redness, (a*); yellowness, (b*). Each loin piece was cut in slices (2.5 cm tick) and the colour of three slices was measured in the sample of each analytical point. Before each series of measurements, the instrument was adjusted using a white ceramic tile. Lipid oxidation was assessed in triplicate by the 2-thiobarbituric acid (TBARS) method of Vyncke (1975) with a modification consisting in the fact that samples were incubated at 96 °C in a forced oven (Memmert UFP 600, Schwabach, Germany). Thiobarbituric acid reactive substances values were calculated from a standard curve performed with 1,1,3,3-tetraethoxypropane and expressed as mg malonaldehyde (MDA)/kg sample.

2.2.3. Chemical composition

Moisture, fat, protein (Kjeldahl N × 6.25) and ash were quantified according to the ISO recommended standards 1442:1997 (ISO, 1997), 1443:1973 (ISO, 1973), 937:1978 (ISO, 1978), and 936:1998 (ISO, 1998), respectively. Total chlorides were quantified according to the Carpentier-Vohlard official method (ISO, 1841-1:1996).

2.2.4. Warner–Bratzler and texture profile analysis

Seven meat pieces of 1 × 1 × 2.5 cm (height × width × length) were removed parallel to the muscle fibre direction and were completely cut using a Warner–Bratzler (WB) shear blade with a triangular slot cutting edge (1 mm of thickness). Maximum shear force was obtained using a texture analyser (TA.XTplus, Stable Micro Systems, Vienna Court, UK). Texture profile analysis (TPA) was measured by compressing to 60% with a compression probe of 19.85 cm^2 of surface contact in seven meat pieces of 1 × 1 × 2.5 cm (height × width × length). Force–time curves were recorded at a crosshead speed of 3.33 mm/s and recording speed was also 3.33 mm/s. Hardness (kg), cohesiveness (unitless), springiness (mm), gumminess (kg) and chewiness (kg * mm) were obtained. These parameters were obtained using the available computer software [Texture Exponent 32 (version 1.0.0.68), Stable Micro Systems, Vienna Court, UK].

2.2.5. Free fatty acid

Total intramuscular lipids were extracted from 5 g of ground loin sample, according to Folch, Lees, and Stanley (1957) procedure. Free fatty acids were separated using NH_2-aminopropyl mini-columns as described by García-Regueiro, Gilbert, and Díaz (1994). Fifty milligrammes of the extracted lipids was transesterified with a solution of boron trifluoride (14%) in methanol, as described by Carreau and Dubacq (1978) and the FAMEs were stored at −80 °C until chromatographic analysis.

Separation and quantification of FAMEs 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, with the column head pressure set at 35.56 psi. One 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, as internal standard that 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/100 g of fat.

2.2.6. Volatile compound analysis

For volatile compound analysis, 3 g of minced foal loin was weighed into a 20 mL headspace vial and sealed with a PTFE-faced silicone septum (Supelco, Bellefonte, PA, USA). A SPME device (Supelco, Bellefonte, PA, USA) containing a fused-silica fibre (10 mm length) coated with a 50/30 μm layer of DVD/CAR/PDMS (Divinylbenzene/Carboxen/Polydimethylsiloxane) was used. The vial was left at 35 °C in a thermo block (Memmert model 100–800, Schwabach, Germany) for 15 min to