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Effect of chemical composition and high pressure processing on the volatile fraction of Serrano dry-cured ham

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2-Propanol (PubChem CID: 3776)
1-Pentanol (PubChem CID: 6276)
2-Methylbutanal (PubChem CID: 7284)
2-Butoxyethanol (PubChem CID: 8133)
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ABSTRACT

The volatile fraction of 30 Serrano dry-cured hams with different salt and intramuscular fat contents was investigated. In addition, the effect of high pressure processing (HPP) at 600 MPa for 6 min at 21 °C on the volatile compounds of those hams was studied. One hundred volatile compounds were identified and their levels subjected to analysis of variance with ham chemical composition (a_w, salt content, intramuscular fat content and salt in lean ratio) and HPP treatment as main effects. Chemical composition mainly affected the relative abundance of acids, alcohols, branched-chain aldehydes, ketones, benzene compounds, sulfur compounds and some miscellaneous compounds. Salt content and fat content influenced a greater number of volatile compounds than a_w. High pressure processing had a significant effect on only 8 volatile compounds, with higher levels of methanethiol and sulfur dioxide in HPP-treated samples and higher levels of ethyl acetate, ethyl butanoate, ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, dimethyl disulfide and dimethyl trisulfide in control untreated samples.

1. Introduction

Serrano ham is a traditional Spanish dry-cured meat product, highly appreciated worldwide. Approximately 18 million Serrano hams are annually produced. Its manufacturing process begins with a salting step, during which salt and other curing ingredients (nitrate and/or nitrite) and additives (ascorbic acid) slowly diffuse into the meat, followed by brushing or washing of hams to remove the excess of salt, a post-salting step and a ripening or drying stage. The whole process usually lasts from 7 to 12 months.

Aroma, one of the most important sensory characteristics of dry-cured ham, is associated with its volatile composition. The volatile fraction of Serrano ham is mainly composed of aldehydes, ketones, alcohols, hydrocarbons, lactones and esters (Flores, Grimm, Toldrá, & Spanier, 1997; Sabio, Vidal-Aragón, Bernalte, & Gata, 1998; Toldrá & Flores, 1998). The presence of most of these volatile compounds in other dry-cured ham varieties has been reported (Barbieri et al., 1992; Berdagué, Denoyer, Le Quéré, & Semon, 1991; Carrapiso, Ventanas, & García, 2002; García et al., 1991; Luna, Aparicio, & García-González, 2006). The complex biochemical reactions that take place through the ripening period are responsible for the characteristic aroma and volatile composition of each of those dry-cured ham varieties. Proteolysis and lipolysis generate peptides, free amino acids and free fatty acids that contribute to the flavor and aroma of the product. These reactions are mainly due to endogenous enzymes, with minor contribution from enzymes of microbial origin. Lipid oxidation and further interaction of the resulting compounds with proteins, peptides and free amino acids, as well as Strecker degradation of free amino acids and Maillard reactions, are responsible for the generation of most of the volatile compounds found in dry-cured ham (Toldrá & Flores, 1998; Ventanas et al., 1992).

Salting of ham plays an important role in the manufacturing process, since NaCl contributes to microbial stability through the reduction of a_w, enhances protein solubilization, affects proteolysis, lipolysis and lipid

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oxidation, improves product texture and contributes directly to flavor (Toldrá & Flores, 1998). However, the relatively high NaCl and fat contents of Serrano ham fail to meet the consumers' demand for healthier food products with lower salt and fat contents. Salt content of ham may be reduced, but lower NaCl concentration increases microbial risk and may cause technological problems. Thus, dry-cured hams of low salt content showed more pronounced rancid, fatty and buttery aroma notes than hams of high salt content (Coutron-Gambotti, Gandemer, Rousset, Maestrini, & Casabianca, 1999). Iberian ham of 6% salt content was drier, harder and more fibrous than ham of 3% salt content, but the effect of salt content on aroma traits was not significant (Andrés, Cava, Ventanas, Muriel, & Ruiz, 2004b) and the differences in salt content (3% or 6%) hardly influenced the levels of volatile compounds, except for 2-pentylfuran (Andres, Cava, Ventanas, Muriel, & Ruiz, 2007). A lower fat content would negatively affect the sensory characteristics of dry-cured ham, since lipids are the substrates for volatile compound formation through chemical reactions (Toldrá & Flores, 1998). Fat not only acts as a reservoir for certain volatile compounds coming from the diet (Ventanas, Estevez, Andrés, & Ruiz, 2008), but also affects taste perception of sweet, salty, sour and bitter stimuli (Lynch, Liu, Mela, & MacFie, 1993) and could influence volatile compound release to the mouth, by retaining in particular non-polar compounds (Ventanas et al., 2008). The influence of salt and fat contents on the perception of flavor and texture in dry-cured hams depends on the variety, being more marked for Iberian ham than for Serrano ham (Lorido, Estévez, Ventanas, & Ventanas, 2015).

High pressure processing (HPP) is a non-thermal technology with a minimal impact on the nutritional and sensory characteristics of meat and meat products (Cheftel & Culioli, 1997). For this reason, HPP is being widely used in the meat industry to eliminate pathogens and spoilage microorganisms, thus ensuring product safety and increasing its shelf-life (Garriga, Grèbol, Aymerich, Monfort, & Hugas, 2004).

The effect of HPP on the volatile fraction of meat products greatly depends on treatment conditions, particularly the pressure level, and on the compositional characteristics of the product (Rivas-Cañedo, Juez-Ojeda, Nuñez, & Fernández-García, 2011). There is little information available on the effect of HPP on the volatile fraction of dry-cured ham. Lower levels of nonane, decane, undecane, 2-undecene, dodecane, ethyl pentanoate and benzaldehyde and higher levels of 2-heptanone were reported in HPP-treated Serrano ham, independently of the packaging material, while 17 volatile compounds increased and 19 compounds decreased after HPP, depending on the packaging material (Rivas-Cañedo, Fernández-García, & Nuñez, 2009a). Enhanced lipid oxidation and protein oxidation were observed in Iberian ham treated at 600 MPa, with a more pronounced effect on ham slices than on non-sliced samples (Fuentes, Ventanas, Morcuende, Estévez, & Ventanas, 2010). Taking into account these results, interactions between HPP treatment and ham characteristics, including its chemical composition, are to be expected. However, the effect of HPP on the volatile compounds of Serrano dry-cured ham with different salt and fat contents has not been studied, to our knowledge.

The objective of the present work was to investigate the influence of Serrano ham chemical composition on the formation of volatile compounds during ripening and to elucidate the changes caused by HPP treatment in the volatile fraction of Serrano hams of different chemical compositions.

2. Materials and methods

2.1. Selection and manufacture of Serrano hams

Manufacture of Serrano hams was carried out at the Institute of Food and Agricultural Research and Technology (IRTA, Monells, Spain). Thirty green hams were selected at commercial slaughterhouses from animals of different genotypes in order to obtain a wide range of fat contents. Twenty-one hams were from Large White × Landrace animals and nine hams from animals with a minimum of 50% Duroc breed. Fat content of entire hams was determined using magnetic resonance sensor technology (Jmp Ingenieros, Sotés, Spain). Homogeneous hams in terms of weight and pH were used in this study. Average weight of hams was 11.77 kg (SD, 0.66 kg) whereas the pH in the semimembranosus muscle at 24 h post-mortem ranged from 5.4 to 5.9. Hams were manually rubbed with the following mixture (per kg of raw ham): 10 g NaCl, 1.0 g dextrose, 0.5 g ascorbic acid, 0.15 g KNO_3 and 0.15 g $NaNO_2$. Afterwards, hams were held with excess of salt at 3 ± 2 °C and 85 ± 5% RH. In order to obtain a wide range of salt and fat contents, 0.6 to 1.5 days of salting per kg of raw ham were applied and hams of different fat contents were selected for each of the salting times, which ranged from 7 to 15 days. After salting, hams were washed with cold water, weighed and hung in a cold room at 3 °C and 75–80% RH to rest. Temperature was progressively increased up to 20 °C during ripening. The process was finished when a total weight loss of 36% was achieved.

2.2. Sampling and high pressure processing

Two slices (approximately 150 g) from the cushion (mainly composed of the *Biceps femoris*, *Semimembranosus* and *Semitendinosus* muscles) were obtained from each ham and individually vacuum-packaged. One of the slices was HPP-treated at 600 MPa for 6 min at 21 °C (pressure build up time, 2.5 min; pressure release time < 2 s) in a 120 L capacity Wave 6000 equipment (Hiperbaric, Burgos, Spain) at IRTA (Monells, Spain) whereas the other slice served as untreated control. Ham slices were held at 4 °C for 3 days and kept at −35 °C at our laboratory until analysis, which took place within 1 month of sampling.

2.3. Physicochemical determinations

Ham homogenates, approximately 50 g in weight and representative of the whole slice, were obtained using a mechanical grinder (IKA Labortechnik, Staufen, Germany). Chloride content was determined by the Volhard method (AOAC, 2000) and intramuscular fat content after extraction with chloroform–methanol (Folch, Lees, & Sloane-Stanley, 1957) on representative subsamples of the homogenate, approximately 3 g in weight. Water activity (a_w) was measured using an AquaLab Series 3-equipment (Decagon Devices, Inc., Pullman, WA, USA), according to the manufacturer instructions. Analyses were performed in triplicate.

2.4. Analysis of volatile compounds

Volatile compounds were extracted by solid-phase microextraction (SPME) and analyzed by gas chromatography–mass spectrometry (GC–MS) (HP 6890-MSD HP 5973, Agilent, Palo Alto, CA, USA). Fifteen grams of Serrano ham, trimmed of subcutaneous fat and representative of the whole ham slice, were homogenized in a mechanical grinder with 15 g of anhydrous Na_2SO_4 (Merck, Darmstadt, Germany) and 30 μL of an aqueous solution of 534 mg/L cyclohexanone (Sigma-Aldrich, Alcobendas, Spain) added as internal standard. A 40 mL headspace glass vial was filled with 10 g of the mixture, sealed with a polytetrafluoroethylene (PTFE) faced silicone septum and introduced in a thermostatic bath at 35 °C (D3 model, Haake, Berlin, Germany) for both equilibration and extraction phases (1 h each). An SPME manual holder equipped with a 2 cm × 50/30 μm StableFlex Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) coated fiber (Supelco, Bellefonte, PA, USA) was inserted through the PTFE septum for headspace extraction. Desorption into the GC injection port took place at 260 °C for 10 min in splitless mode. Chromatographic separation was carried out in a Zebron 100% polyethylene glycol capillary column (60 m long; 0.25 mm internal diameter; 0.50 μm film thickness; ZB-WAXplus, Phenomenex, Torrance, CA, USA) with 1 mL/min helium flow, with the following temperature program: 16 min at 45 °C, a first ramp at 4 °C/min to 110 °C, 9 min at 110 °C, a second ramp at 15 °C/min to 230 °C and 3 min at 230 °C, a final ramp at 10 °C/min