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Influence of physicochemical characteristics and high pressure processing on the volatile fraction of Iberian dry-cured ham

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ABSTRACT

The volatile fraction of 30 Iberian dry-cured hams of different physicochemical characteristics and the effect of high pressure processing (HPP) at 600 MPa on volatile compounds were investigated. According to the analysis of variance carried out on the levels of 122 volatile compounds, intramuscular fat content influenced the levels of 8 benzene compounds, 5 carboxylic acids, 2 ketones, 2 furanones, 1 alcohol, 1 aldehyde and 1 sulfur compound, salt concentration influenced the levels of 1 aldehyde and 1 ketone, salt-in-lean ratio had no effect on volatile compounds, and water activity influenced the levels of 3 sulfur compounds, 1 alcohol and 1 aldehyde. HPP-treated samples of Iberian ham had higher levels of 4 compounds and lower levels of 31 compounds than untreated samples. A higher influence of HPP treatment on volatile compounds than physicochemical characteristics was observed for Iberian ham. Therefore, HPP treatment conditions should be optimized in order to diminish its possible effect on Iberian ham odor and aroma characteristics.

1. Introduction

Iberian dry-cured ham is manufactured in Spain from the hind legs of Iberian breed pigs. The traditional process consists in the addition of curing salts, which diffuse into the ham during the post-salting stage, followed by a long ripening-dehydration stage lasting up to 48 months during which its flavor and aroma develop.

The unique distinctive aroma of Iberian ham is partly derived from the rich lipid fraction characterizing the meat of this autochthonous animal breed (Andrés, Cava, Martin, Ventanas, & Ruiz, 2005). In dry-cured ham, free fatty acids are generated from the lipids in muscle and adipose tissue while small peptides and free amino acids derive from muscle proteins (Toldrá & Flores, 1998; Zhang, Jin, Wang, & Zhang, 2011). Part of these intermediate compounds is transformed into volatile compounds through lipid oxidation, Maillard reactions, Strecker degradation and other reactions (García et al., 1991; López et al., 1992; Zhang, Zhen, Zhang, Zeng, & Zhou, 2009). Chemical composition of meat, added curing salts and processing conditions affect the formation and stability of volatile compounds in dry-cured ham.

Current consumer trends lead towards the production of low-salt low-fat dry-cured ham. However, changes in the chemical composition of ham could impair its microbial stability, affect its shelf-life and influence its volatile fraction (Armenteros, Toldrá, Aristoy, Ventanas, & Estévez, 2012; Blesa et al., 2008). Proteolysis, lipolysis and lipid oxidation phenomena might be influenced by variations in physicochemical characteristics such as salt concentration, water activity (a_w) and fat content and the generation of aroma compounds might thus be altered. Salt concentration influenced the formation of some volatile compounds in Iberian ham (Andrés, Cava, Ventanas, Muriel, & Ruiz, 2004a; Andrés, Cava, Ventanas, Muriel, & Ruiz, 2007). In dry-cured turkey ham, salt concentration correlated positively with total aldehydes content and negatively with alcohols, ketones and alkanes contents (Wang, Jin, Zhang, Ahn, & Zhang, 2012). Regarding the influence of fat content on the formation of volatile compounds, different volatile profiles were found between muscles and subcutaneous fat of dry-cured hams (Sánchez-Peña, Luna, García-González, & Aparicio, 2005). A higher abundance of volatile compounds was found in Iberian dry-cured loins of high intramuscular fat content than in those of low intramuscular fat content (Ventanas, Estevez, Andrés, & Ruiz, 2008). Also, the levels of 16 out of 39 volatile compounds differed between Iberian hams from pigs with different fatty acid composition of the intramuscular fat (Carrapiso et al., 2015).

Ready-to-eat sliced meat products have gained worldwide consumer acceptance. However, pathogens and spoilage microorganisms may reach the product during the deboning, slicing and packaging of ready-to eat dry-cured ham, affecting its safety and shelf-life. For this reason, high pressure processing (HPP) is widely used at the meat industry to eliminate undesirable contaminating microorganisms (Clariana et al.,

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2011; Garriga, Grèbol, Aymerich, Monfort, & Hugas, 2004).

HPP treatment is also known to affect the volatile compounds of Serrano dry-cured ham (Rivas-Cañedo, Fernández-García, & Nuñez, 2009) and the lipid oxidation phenomena of Iberian dry-cured ham (Andrés, Adamsen, Møller, Ruiz, & Skibsted, 2006; Andrés, Møller, Adamsen, & Skibsted, 2004b; Fuentes, Ventanas, Morcuende, Estévez, & Ventanas, 2010).

The effect of HPP on the volatile fraction of Serrano hams of different chemical composition was recently studied (Martínez-Onandi, Rivas-Cañedo, Nuñez, & Picon, 2016). However, information on the effect of HPP treatment on the volatile compounds of Iberian ham is scarce. To our knowledge, only the effect of HPP treatment on the volatile aldehydes of Iberian ham has been reported (Fuentes et al., 2010). Information on the influence of the physicochemical characteristics of Iberian ham on its volatile fraction is also limited. Iberian ham has higher intramuscular fat content, lower salt concentration and salt-in-lean ratio, and higher a_w than Serrano ham, compositional characteristics which might influence the formation of volatile compounds during manufacture and ripening as well as the effect of HPP treatment. The objective of the present study was to investigate the influence of those physicochemical characteristics and HPP treatment on the volatile fraction of Iberian dry-cured ham.

2. Material and methods

2.1. Selection of Iberian hams

Thirty Iberian hams from 50% Iberian × Duroc animals, manufactured and ripened at a processing plant in Extremadura (Spain), were selected at the Institute of Food and Agricultural Research (IRTA, Monells, Spain) on the basis of their subcutaneous fat thickness and their salt content estimated by means of computed tomography (Santos-Garcés, Gou, García-Gil, Arnau, & Fulladosa, 2010) as previously described (Martínez-Onandi et al., 2016).

2.2. Sampling and high pressure processing

Two slices (150–200 g in weight) obtained from the cushion of each ham (mainly composed of the *Biceps femoris*, *Semimembranosus* and *Semitendinosus* muscles) were individually vacuum-packaged in polyamide + polyethylene bags (Mobepack, Salamanca, Spain). One of the slices was HPP-treated at 600 MPa for 6 min at 21 °C as previously described (Martínez-Onandi et al., 2016). The other slice served as untreated control. Both slices were held at 4 °C for 3 days and afterwards kept at − 35 °C until analysis, for not > 30 days.

2.3. Physicochemical determinations

Prior to analysis, a representative 100 g portion of each ham slice was minced by means of a mechanical grinder (IKA Labortechnik, Staufen, Germany). Intramuscular fat content was determined by the Folch method (Folch, Lees, & Slone Stanley, 1957), chloride content by the Volhard method (AOAC, 2000) and a_w by means of an AquaLab Series 3 equipment (Decagon Devices Inc., Pullman, WA, USA). All analyses were performed in triplicate.

2.4. Analysis of volatile compounds

A representative 15 g portion of each ham slice was homogenized in a mechanical grinder (IKA Labortechnik) 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) as internal standard. Volatile compounds were extracted by solid-phase microextraction (SPME), analyzed by gas chromatography–mass spectrometry (GC–MS) and identified as previously described (Martínez-Onandi et al., 2016). Semi-quantitative determination of volatile compounds and expression of results was as indicated by those authors. Each ham was analyzed in triplicate.

2.5. Statistical analysis

Experimental data were analyzed using the SPSS 19.0 statistical package (SPSS Inc., Chicago, IL, USA). Three groups of hams of low, medium and high values for each of the physicochemical characteristics were differentiated by using the mean ± 0.5 standard deviations (SD) criterion (Martínez-Onandi et al., 2016). Analysis of variance was carried out with each of the physicochemical characteristics as main effect and ham as random effect or with HPP treatment as main effect and ham as fixed effect. Tukey's test with the significance assigned at $P < 0.05$ was used for the comparison of means. Principal component analysis (PCA), with Varimax rotation, was carried out on total levels of chemical groups of volatile compounds, levels of individual volatile compounds, physicochemical characteristics and HPP treatment.

3. Results and discussion

3.1. Iberian ham volatile compounds

Volatile compounds detected in the present study included 10 carboxylic acids, 18 alcohols, 11 aldehydes, 18 ketones, 10 esters, 13 alkanes, 23 benzene compounds, 5 sulfur compounds, 4 furanes, 5 furanones, 3 pyrazines and 2 miscellaneous compounds (Table 1). These 122 volatile compounds were found in all control and HPP-treated samples.

The volatile fraction of Iberian ham has been the subject of previous studies (García et al., 1991; Ruiz, Cava, Ventanas, & Jensen, 1998). Factors such as animal genotype (Ramírez & Cava, 2007), animal diet (López et al., 1992), ham ripening time (Ruiz, Ventanas, Cava, Andrés, & García, 1999), muscle (Sánchez-Peña et al., 2005), and anatomical location (Narváez-Rivas, Gallardo, Ríos, & León-Camacho, 2010; Timón, Ventanas, Carrapiso, Jurado, & García, 2001) influenced Iberian ham volatile fraction. The number of volatile compounds detected in the above cited studies on Iberian ham ranged from 55 to 109, but the total number of volatile compounds found in the literature on Iberian ham rises to 411 (Narváez-Rivas, Gallardo, & León-Camacho, 2012).

Carboxylic acids reported in previous works on Iberian ham ranged from 1 (García et al., 1991) to 6 (Ramírez & Cava, 2007) in number, alcohols from 7 (Ruíz et al., 1999) to 14 (Sabio, Vidal-Aragón, Bernalte, & Gata, 1998), aldehydes from 12 (Ramírez & Cava, 2007) to 19 (Ruiz et al., 1998; Timón et al., 2001), ketones from 3 (López et al., 1992) to 15 (Sabio et al., 1998), esters from 2 (Ramírez & Cava, 2007) to 10 (Sabio et al., 1998), sulfur compounds from 1 (García et al., 1991) to 8 (Sabio et al., 1998), and nitrogen compounds from 2 (Ruiz, Ventanas, Cava, Andrés, & García, 1999) to 5 (Ruiz et al., 1998). The main reason for those differences in the number of volatile compounds would probably be the analytical procedure used, although ham characteristics due to feeding of animals and manufacturing and ripening conditions might also be involved.

In the present study, compounds presumably derived from lipid oxidation (linear carboxylic acids, linear alcohols, linear aldehydes, linear ketones, and hydrocarbons) accounted for 75.0% of the volatile fraction of untreated Iberian ham, compounds derived from Maillard reactions (branched-chain carboxylic acids, branched-chain alcohols, branched-chain aldehydes, branched-chain ketones, nitrogen compounds and sulfur compounds) for 18.1%, and compounds of microbial or unknown origin (acetic acid and esters) for 6.9% (data not shown). In a previous work on Iberian ham the results obtained were similar, with 81.6% of its volatile fraction considered to derive from lipid oxidation, 12.7% from Maillard reactions and 5.7% from microbial or unknown origin (Ramírez & Cava, 2007).

Not all the volatile compounds present in Iberian ham are equally