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Determination of volatile compounds and quality parameters of traditional Istrian dry-cured ham

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ABSTRACT

The aim of this work was to determine the characteristics of Istrian dry-cured ham by instrumental methods and sensory analysis. The aroma-active compounds of Istrian dry-cured ham from 2010 and 2012 were investigated by using headspace-solid phase microextraction (SPME) and gas chromatography–mass spectrometry (GC–MS). Samples of *biceps femoris* were also evaluated by measuring physical and chemical characteristics. 92 volatile aroma compounds of Istrian dry-cured ham were found. Volatile compounds belonged to several chemical groups: aldehydes (51.4; 51.3%), terpenes (16.5; 16.4%), alcohols (15.5; 13.2%), ketones (8.6; 7.4%), alkanes (3.8; 5.7%), esters (1.3; 1.6%), aromatic hydrocarbons (0.8; 3.9%) and acids (0.6; 0.9%). Principal component analysis (PCA) showed that fat content, tenderness and melting texture were positively correlated. Terpenes were strongly correlated with flavour of added spices. Sweet taste and the presence of esters were positively correlated as well as negative odour, raw meat flavour and water content.

1. Introduction

Istrian dry-cured ham is a typical dry-cured Croatian product and the tradition in its production makes it different from all other Mediterranean dry-cured hams. Croatia has recognised the importance of its unique autochthonous products, and Istrian dry-cured ham has received the designation of origin according to EU standards in 2011. The ham is produced from Landrace, Large White and Duroc pigs and their crossbreeds with a minimum live weight at slaughter of 160 kg. Istrian ham is processed with pelvic bones, and skin and subcutaneous fatty tissue are removed. The processing of Istrian dry-cured ham involves four phases: salting and flavouring with garlic, pepper and laurel, pressing, drying and ripening. The process takes between 12 and 18 months (Comi, Orlic, Redzepovic, Urso, & Iacumin, 2004).

The aroma is a key attribute that impacts the overall acceptance of dry-cured hams and is due to the presence of volatile compounds, most of them produced by lipolysis and proteolysis (Toldrá, 1998) during the maturation process (Flores, Grimm, Toldrá, & Spanier, 1997). The aroma is markedly affected by raw material, processing techniques, and ageing time (Pham et al., 2008). Therefore, an understanding of the dry-cured ham aroma should include the identification and quantification of its volatiles. The flavour and aroma of dry-cured ham can also be determined by sensory descriptive analysis and the composition of aroma impact compounds. Several studies have reported information on the volatile composition of various kinds of dry-cured hams, which are very different in their aroma, such as Corsican, Iberian, Serrano and Parma hams (Berdagué, Denoyer, Le Quéré, & Semon, 1991; Bolzoni, Barbieri, & Virgili, 1996; Flores et al., 1997; López et al., 1992; Pastorelli et al., 2003; Timón, Ventanas, Carrapiso, Jurado, & García, 2001), but little information is available for Istrian dry-cured ham.

The objective of this research was to determine volatile compounds in traditional Istrian dry-cured ham using solid phase microextraction (SPME) and gas chromatography–mass spectrometry (GC–MS). Another purpose of this work was to determine the chemical composition and physico-chemical aspects of Istrian dry-cured ham and their possible connection with the formation of volatile compounds. Comparison of the results of sensory analysis, volatile compounds and physico-chemical parameters was also done.

2. Materials and methods

2.1. Traditional production process

Commercially produced Istrian dry-cured hams were obtained from Landrace, Large White and Duroc pigs and their crossbreeds with a minimum live weight at slaughter of 160 kg. It is produced with pelvic bones and without skin and the subcutaneous adipose tissue. Dry salting of hams is conducted using sea salt with an addition of ground black pepper, garlic and laurel. Quantities of added spices vary between manufacturers. Salting is conducted in cooling chambers at a temperature of 0–5 °C and a relative humidity of 80–90%, for a period of 21 days, including the pressing for 7 last days. Prior to drying, all hams are sprinkled with a mixture of herbs. Drying is done in drying chambers with controlled microclimatic conditions (air circulation—10–20 cm/s; temperature—12–16 °C; humidity gradually reduced

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from 90 to 70%) lasting 158 days. Ripening of hams takes place in cellars with a stable microclimate and the possibility for complete darkness, and an air temperature which does not exceed 18 °C in summer (between 12 and 18 °C year round) and relative air humidity between 65 and 75% until they become 12–18 months old (Marušić, Petrović, Vidaček, Petrak, and Medić (2011)).

2.2. Ham sampling

Samples of *biceps femoris* of traditional Istrian dry-cured ham were obtained from 11 manufacturers in 2010 and from 8 manufacturers in 2012. Samples of *biceps femoris* from each manufacturer were analysed for volatile compounds, chemical composition and physical characteristics. Samples of Istrian dry-cured ham from 2012 were also evaluated by a sensory panel.

2.3. Chemical composition analysis

Fat, protein and ash contents were estimated according to methods recommended by the AOAC (1999). Moisture content and sodium chloride were determined in the *biceps femoris* according to AOAC methods (AOAC, 1984). Two replicates of each sample were analysed and the mean value was used in the data analyses. Water activity of the *biceps femoris* was determined with a precision multi-function measuring instrument, Testo 650 (Testo Inc., New York, USA). Two replicates of each sample were analysed and the mean value was used in the data analyses.

2.3.1. Lipid oxidation by the TBARS test

Oxidation of lipids was assessed by the thiobarbituric acid (TBA) assay which is based on the reaction between TBA and malondialdehyde (MDA) and the production of a coloured pigment, the concentration of which is calculated by measuring the absorbance at 538 nm on three replicates of each sample (Lemon, 1975). The spectrophotometer was a Helios β (Spectronic Unicam, Cambridge, UK). A calibration curve was developed using 0, 0.01, 0.02, 0.03, 0.04 and 0.05 μmol of MDA. TBARS values were expressed as mg of malondialdehyde equivalents/kg dry-cured ham.

2.3.2. Colour instrumental measurement

Colour measurements were carried out with a Minolta CM-3500d (Osaka, Japan) spectrophotometer in the CIELAB space: lightness (L*), redness (a*) and yellowness (b*) (CIE, Commission Internationale de l'Eclairage, 1976). Each sample of *biceps femoris* was analysed in ten replicates, avoiding regions with excess fat to achieve representative measurements of the lean colour.

2.4. Analysis of volatile compounds

Analyses were carried out by extraction of volatile compounds above the samples on SPME fibre and their qualification and quantification on GC/MS by the method as described by Marušić et al. (2011).

30 g of *biceps femoris* muscle slices from dry-cured ham was ground with a commercial grinder. Then dry-cured ham homogenates were prepared by dispersing 5 g of minced muscle slices with 25 mL of distilled water saturated with NaCl in a commercial blender. Ten millilitres of this mixture was placed into 20 mL vials tightly capped with a PTFE septum. A magnetic stirrer was placed into the homogenates for stirring during extraction.

A SPME fibre coated with 2 cm of 50/30 μm DVB/Carboxen/PDMS (Supelco, Bellefonte, PA, USA) was conditioned for 2 min at 240 °C prior to extraction and placed above the sample mixture. Triplicate 20 mL vials were placed in a water bath at 40 °C and extracted for 180 min with stirring. After extraction the SPME fibre was immediately injected to 6890N gas chromatograph coupled to a 5975i mass selective detector (Agilent Technologies, Santa Clara, CA, USA). Capillary column DB-5ms 30 m × 0.25 mm, film thickness 0.25 μm (Agilent Technologies, Santa Clara, CA, USA) was used with helium as a carrier gas at 1.0 mL/min flow rate. The temperature of the injector, used in the splitless mode, was 230 °C and desorption time was 2 min. Temperature programme was at 40 °C, isothermal for 10 min, then rising to 200 °C at a rate of 5 °C/min and then raising to 250 °C at a rate of 20 °C/min. Final temperature was held for 5 min. The transfer line temperature was maintained at 280 °C. The mass spectra were obtained at 70 eV with a rate of 1 scan/s over the m/z range of 50–450.

An in-house mixture of C8–C20 n-alkanes was run under the same chromatographic conditions to calculate the retention indices (RI) of detected compounds. AMDIS 3.2 program version 2.62 was used for identification of components using NIST 2005 version 2.0 spectral library (NIST, Gaithersburg, MD, USA) as well as comparison of obtained retention indices with literature values (Adams, 2001 and in-house library).

2.5. Sensory analysis

Istrian dry-cured hams from eight producers from 2012 were assessed by seven trained panellists who were selected and trained in accordance with international standard (ISO 8586:2012). Panel members were situated in a private red lighted cabinet during sessions.

Samples were individually labelled and were randomly served one at a time. All hams were evaluated in slices from the same anatomical area. In each sensory session, panellists evaluated 2 samples and the sensory evaluation consisted of eight sessions (each sample was evaluated two times). Panellists were asked to indicate point of the scale corresponding to the intensity of their different feelings for each attribute. Sensory attributes were assessed with a 10 point intensity line scale, where 0 = not detected and 9 = extremely strong. All the samples, slices of 1.5 mm thickness, were evaluated at 20–22 °C in sensory panel rooms. About 50 mL of water and 20 g of unsalted bread were provided to assessors between successive ham samples (García-González et al., 2006).

Fourteen traits related to sensory characteristics of dry-cured hams (Table 1) were evaluated by the quantitative-descriptive analysis method. The traits were grouped into appearance (red colour, marbling, and white crystals), odour (positive odour, negative odour, odour typical for dry-cured ham), texture (tenderness, melting texture), taste (salty and sweet taste), flavour (flavour of added spices, raw meat flavour) and acceptability (number of positive characteristics, aroma duration).

2.6. Statistical analyses

One-way ANOVA was carried out for physical and chemical data using the SPSS 12.0 computer programme. Statistical significance was set at $P < 0.05$. Student's t-test was used to determine whether there

Table 1
Codes, sensory attributes and mean intensities of the sensory attributes in Istrian dry-cured ham from 2012.

Code	Sensory attributes	Istrian dry-cured ham
A1	Red colour	7.2 ± 1.3
A2	Marbling	5.1 ± 2.6
A3	White crystals	1.1 ± 1.6
A4	Positive odour	7.3 ± 1.1
A5	Negative odour	2.0 ± 1.6
A6	Odour typical for dry-cured ham	7.5 ± 1.1
A7	Tenderness	7.0 ± 1.2
A8	Melting texture	6.5 ± 1.4
A9	Salty taste	6.5 ± 0.8
A10	Sweet taste	2.3 ± 2.3
A11	Flavour of added spices	4.0 ± 1.4
A12	Raw meat flavour	0.7 ± 1.2
A13	Number of positive characteristics	6.9 ± 1.1
A14	Aroma duration	6.9 ± 1.2