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# Differentiation of dry-cured hams from different processing methods by means of volatile compounds, physico-chemical and sensory analysis

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ABSTRACT

The aim of this study was to characterize dry-cured hams from four different processing methods (differences in primary leg treatment, salting and smoking phase). Volatile compounds were isolated by using headspace-solid phase microextraction and analysed by gas chromatography/mass spectrometry (GC/MS). Samples were also evaluated by sensory and physico-chemical characteristics (moisture, protein, fat and NaCl content, $a_w$, colour). 149 volatile compounds of dry-cured hams were identified and 15 of them were quantified. Identified volatile compounds belonged to several classes of chemical: 25 aldehydes, 18 phenols, 12 alcohols, 16 terpenes, 27 aromatic hydrocarbons, 18 aliphatic hydrocarbons, 17 ketones, 9 esters and 7 acids. Most abundant volatiles in ham samples were aldehydes (34.46–49.78%). Principal component analysis showed a good separation among groups. Smoked dry-cured hams showed a higher content of phenols, aromatic hydrocarbons, and acids and were characterized by smoky aroma, while non-smoked dry-cured hams showed higher content of terpenes, ketones, alcohols, esters, aliphatic hydrocarbons and were characterized with spicy aroma.

## 1. Introduction

Dry-curing of hams is a traditional process in the Mediterranean region that leads to a product with unique flavour. The dry-cured ham quality is markedly affected by the raw material and stages during the production process (salting, washing, post-salting for salt equalization, smoking and ripening-drying). Therefore, dry-cured ham is a complex product, since the variety of processing technologies and the influence of the raw material (genetic type, feed, rearing system, etc.) contribute to its quality, especially sensory characteristics and volatile compounds (Toldrá, 2004).

The aroma of dry-cured ham is mainly due to the development of volatile compounds as a result of the proteolysis and lipolysis that take place during the ripening stage of the processing. These volatile compounds include aldehydes, alcohols, ketones, carboxylic acids, hydrocarbons and others. Many biochemical changes (lipolysis, proteolysis, oxidation reactions, Strecker degradation and Maillard reactions) take place during the manufacturing and ripening of dry-cured ham and contribute to flavour development (Martínez-Onandi, Rivas-Cañedo, Picon, & Nuñez, 2016). Lipolysis and proteolysis are the main biochemical reactions involved in the generation of these compounds, producing a wide range of volatiles and precursors. Several studies have reported sensory attributes and volatile compounds in qualifying dry-cured hams especially in Spanish, Italian and French dry-cured hams (Flores, Aristoy, Spanier, & Toldrá, 1997; García-González, Tena, Aparicio-Ruiz, & Morales, 2008; Laureati et al., 2014) and also Slovenian dry-cured ham (Pugliese et al., 2015). Also dry-cured ham has been largely studied for its physico-chemical and sensory characteristics depending on different processing technologies (Andrés, Cava, Ventanas, Thovar, & Ruiz, 2004; Costa-Corredor, Serra, Arnau, & Gou, 2009; Flores et al., 2006; Pugliese et al., 2015). However there are only few studies regarding how different technologies (especially usage of spices and smoking in dry-cured ham production) affect dry-cured ham quality.

The samples used in this study were four types of Croatian dry-cured hams of which three have Protected Geographical Indications (PGI) (Krčki, Dalmatinski and Drniški pršut) and one dry-cured ham with Protected Designation of Origin (PDO) (Istarski pršut). Only few studies on Istarski and Dalmatinski pršut have been previously reported (Jerković, Mastelić, & Tartaglia, 2007; Marušić, Petrović, Vidaček, Petrak, & Medić, 2011; Marušić Radovčić, Vidaček, Janči, & Medić, 2016; Marušić, Vidaček, Janči, Petrak, & Medić, 2014), There is no data on characteristics of Krčki and Drniški pršut which have PGI. So the aim of this work is to determine how dry-cured ham production process of these four types of dry-cured ham affects the physico-chemical composition, volatile compounds and sensory characteristics.

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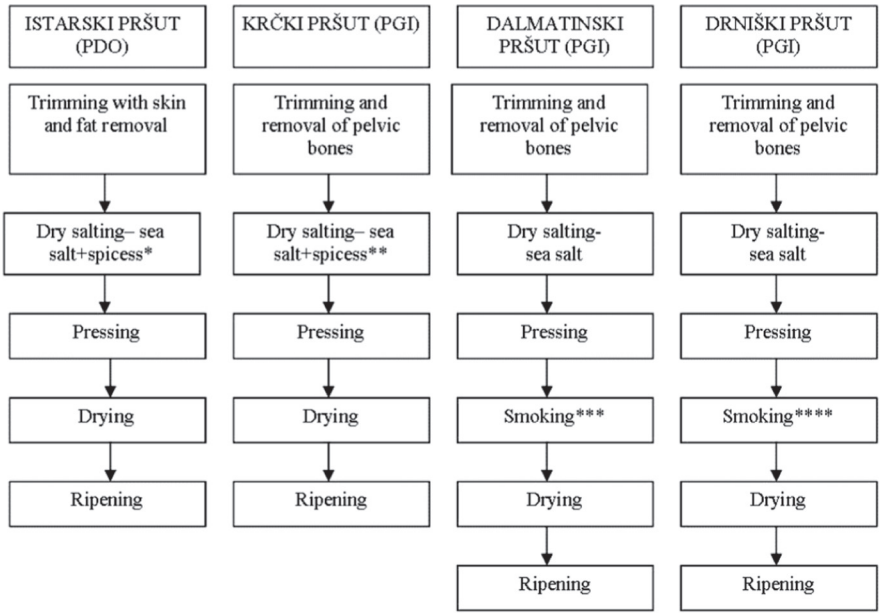


**Fig. 1.** Process flow diagrams for production of Istarski, Krčki, Dalmatinski and Drniški pršut.
*Sea salt + ground black pepper (*Piper nigru*) + laurel (*Laurus nobilis*) and rosemary (*Rosmarinus officinalis*).
**Sea salt + laurel (*Laurus nobilis*) and rosemary (*Rosmarinus officinalis*).
***Cold smoking obtained by burning hardwoods or sawdust of beech (*Fagus* sp.), oak (*Quercus* sp.) or hornbeam (*Carpinus* sp.)
****Cold smoking obtained by burning hardwoods of beech (*Fagus* sp.), hornbeam (*Carpinus* sp.) and local plants like dry twigs spruce (*Juniperus communis*), wood and shell almonds (*Amygdalus communis*) and dry immortelle (*Helichrysum arenarium*).

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## 2. Materials and methods

### 2.1. Dry-cured hams

The research was carried out on 24 dry-cured hams, obtained by processing 24 pig thighs following the different PDO (Istarski pršut) and PGI (Krčki, Dalmatinski and Drniški pršut) specifications. Pigs belonged to Duroc × (Yorksire × Landrace) pig breed and were reared and fed with standard diet under the same conditions. Slaughtering weight of the pigs was 160 kg. Dry-cured hams were prepared according to the traditional processing procedures without any additives such as nitrites or ascorbic acid. Processing of dry-cured hams was performed following the four protocols, as summarized in Fig. 1. Brifely, Dalmatinski, Drniški and Krčki pršut are produced with pelvic bones and with skin and the subcutaneous adipose tissue while during production of Istarski pršut skin and the subcutaneous adipose tissue are removed. Dry salting of hams is conducted using sea salt or combination of sea salt and spices. In the production of Dalmatinski and Drniški pršut only sea salt is used. Curing mixture used for Istarski pršut are: sea salt, ground black pepper, laurel and rosemary; while sea salt, laurel and rosemary in Krčki pršut. Salting is conducted in cooling chambers at a temperature of 0–5 °C and a relative humidity of 80–90%, for a period of up to 1 month depending of the weight of raw ham. Salting phase is followed by pressing phase in the duration of 7–10 days. Drying of all four types of investigated dry-cured hams is done in drying chambers with controlled microclimatic conditions ($T$ 12–16 °C; RH gradually reduced from 90 to 70%). During drying phase Dalmatinski and Drniški pršut are cold smoked ($T < 22$ °C) for 20 days. The hams are further moved to a cellar for ripening at mild temperatures (12–15 °C) and RH 65–75%. At the end of ripening (17 months Istarski pršut, 15 months Krčki, Dalmatinski and Drniški pršut), six hams of four different processing methods were sampled for analysis.

Samples of *biceps femoris* were coded, vacuum packed, frozen and stored at − 18 °C. *prior* to analysis, samples were thawed for 24 h at 4 °C and were analysed for volatile compounds and physico-chemical composition. Sensory analysis was performed on dry-cured samples after sampling.

### 2.2. Chemical composition analysis

Moisture content and chloride content were determined using official methods (AOAC, 2000). Fat content was estimated according to methods recommended by the AOAC (1999). Protein content was determined by Kjeldahl method (ISO 937, 1978). Water activity ($a_w$) was measured using an aw-meter LabMaster-aw (Novasina), according to the manufacturer instructions. Two replicates of each sample were analysed and the mean value was used in the data analyses.

### 2.3. Colour instrumental measurement

Colour measurements were carried out with a Minolta CM-700d (Osaka, Japan) spectrophotometer (illuminant D65, 10° standard observer, 8 mm aperture, with open cone). The L* (lightness), a* (redness), and b* (yellowness) colour was measured (CIE, Commission Internationale de l'Eclairage, 1976). Before analysis spectrophotometer was calibrated with White Calibration Cap CM-A177. 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). Dry-cured ham homogenates were prepared by dispersing 5 g of minced *biceps femoris* 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, 100 μL of 4-methyl-2-pentanol (1.2 mg/kg) (internal standard) was added, tightly capped with a PTFE septum. A magnetic stirrer was placed into the homogenates for stirring during extraction. A