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Application of temperature and ultrasound as corrective measures to decrease the adhesiveness in dry-cured ham. Influence on free amino acid and volatile compound profile

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ABSTRACT

The impact of low temperature treatment and its combination with ultrasound has been evaluated in order to correct texture defects in dry-cured hams. A total of 26 dry-cured hams, classified as high proteolysis index (PI > 36%), were used. From these hams, ten slices from each ham sample were cut, vacuum packed and submitted to three different treatments: control (without treatment), conventional thermal treatments (CV) and thermal treatment assisted by power ultrasound (US). The impact of these treatments on instrumental adhesiveness, free amino acid and volatile compounds profile were assessed. Statistical analysis showed that both US and CV treatments, significantly ($P < .001$) decreased the instrumental adhesiveness of dry-cured hams from 85.27 g for CO to 40.59 and 38.68 g for US and CV groups, respectively.

The total free amino acid content was significantly ($P < .001$) affected by both treatments, presenting higher values the samples from the US group (6691.5 *vs.* 6067.5 *vs.* 5278.2 mg/100 g dry matter for US, CV and CO groups, respectively). No significant differences were observed between US and CV treatments. All the individual free amino acids were influenced by ultrasound and temperature treatments, showing the highest content in sliced dry-cured ham submitted to ultrasounds at 50 °C, except for isoleucine which presented the highest level in samples from CV group. Similarly, significant differences ($P < .05$) were also detected in the total volatile compound content between CO and US groups, with a higher concentration in the CO batch (56,662.84 AU × 10^3/g of dry-cured ham) than in the US treatment (45,848.47 AU × 10^3/g of dry-cured ham), being the values in the CV treatment intermediate (48,497.25 AU × 10^3/g of dry-cured ham). Aldehydes, ethers and esters, carboxylic acids and sulphur compounds were more abundant in the CO group, while CV group showed higher concentrations of ketones, alcohols and nitrogen compounds.

1. Introduction

In terms of economic value, dry-cured ham is the most important meat product in the Spanish market. Nevertheless, its production experienced a gradual reduction during the last years (Ministerio de Agricultura y Pesca, 2017). This may be a consequence of consumer's increasing concern for health. Dry-cured products have been reported to be one of the main sources of dietary salt in Spain, and it is known that sodium is highly related to cardiovascular diseases (WHO, 2012). Consequently, the reduction of salt in dry-cured ham could improve the value of this product by addressing consumer's requirements.

However, negative impact on texture quality due to the reduction of salt in dry-cured meat products has been widely reported (Armenteros, Aristoy, Barat, & Toldrá, 2009; Flores et al., 2006; Lorenzo, Fonseca, et al., 2015). In this regard, excessive proteolysis during dry-cured ham processing may lead to a high instrumental adhesiveness, a high pastiness perception and thus a decrease of consumers' acceptability (López-Pedrouso et al., 2018). In addition, other factors such as properties of fresh pieces (pH, fat level, weight), ripening process and type of muscle have been related to proteolysis index of dry-cured ham (Škrlep et al., 2011). López-Pedrouso et al. (2018) noticed that the determination of instrumental adhesiveness could be a good indicator

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of pastiness level in dry-cured ham. These authors also observed that hams with higher proteolysis indices displayed increased instrumental adhesiveness.

On the other hand, consumer preference highly depends on the sensory properties of slices, which are mainly determined by aroma, taste and texture (Narváez-Rivas, Gallardo, & León-Camacho, 2012). In this regard, aroma of dry-cured ham is due to the presence of many volatile compounds generated by chemical and enzymatic mechanisms during the ripening process (Bermúdez, Franco, Carballo, & Lorenzo, 2015). A great number of volatile compounds has been found in dry-cured ham, including hydrocarbons, ketones, acids, terpenes, ketones, alcohols, nitrogen and sulphur compounds, and others. However, only a limited number of volatile compounds contribute to the overall ham flavor (mainly aldehydes and ketones) (Carrapiso, Ventanas, & García, 2002).

Mild thermal treatments (around 30 °C) during a long time (between 7 and 10 days) have been used to correct the softness and pastiness of dry-cured ham (Gou, Morales, Serra, Guàrdia, & Arnau, 2008; Morales, Arnau, Serra, Guerrero, & Gou, 2008). However, these treatments are not useful for the meat industries because they require a long processing time which could affect to sensorial characteristics (mainly aroma and color) of dry-cured hams. Thus, in order to avoid these defects and improve the final quality of dry-cured ham, new corrective measures that produce a more homogeneous increase of temperature of the ham need to be explored. In this regard, the application of ultrasounds (US) treatment could be a suitable alternative to conventional thermal treatment (Önür et al., 2018). In addition, US can induce chemical, biological and mechanical changes in meat and meat products due to cavitations in liquid systems (Kang et al., 2016) and its effect of dry-cured hams has not been previously investigated.

Low-intensity US waves are used to obtain information about the propagation medium, while high-intensity waves, or high-power US, are used to make permanent changes in the medium (Robles-Ozuna & Ochoa-Martínez, 2012). High-intensity US application is based in the elastic deformation of ferroelectric materials caused by the mutual attraction of polarized molecules into an electric field (Raichel, 2006). In addition, Sajas and Gorbatow (1978) considered that ultrasonic intensity is closely related to the appearance and magnitude of US effects. In a previous study, Contreras, Benedito, Bon, and Garcia-Perez (2018) noticed that heating caused an increase in hardness and elasticity of dry-cured ham, whereas the application of US did not modify the texture parameters. However, to date the application of US as a corrective measure for adhesiveness of dry-cured meat products has not been explored.

Previous studies noticed that the structure and the function of protein can be modified by the application of US. Thus, the objective of this study was to evaluate the high-power US combined with moderate thermal treatments as a non-invasive intervention strategy to decrease the adhesiveness of sliced dry-cured ham, as well as the assessment of the effects of these treatments on the free amino acid and volatile compound contents of ham samples.

2. Materials and methods

2.1. Samples

For this study, a total of 26 dry-cured hams, classified as having a high proteolysis index (PI > 36%) were used. Hams were manufactured according the process reported by Fulladosa et al. (2018). At the end of the process, hams were cut and boned and the cushion part containing the *Biceps femoris* muscle was excised and sampled. Ten slices from each ham sample were vacuum packed and submitted to three different treatments: control (without treatment), conventional thermal treatments (CV) and thermal treatment assisted by power ultrasound (US).

a) Thermal treatments assisted by power ultrasound (US), where ultrasound was only applied during the heating stage, which was defined as the time needed to reach in the centre of the slice a temperature 5 °C below that in the heating medium, measured using a thermocouple. Thus, average ultrasonic treatment time was of 7.5 min. Finally, samples were kept in a water bath (50 °C) to complete 5 h of treatment. This heating temperature and time were chosen to avoid the appearance of cooking flavours in the ham, as found in preliminary experiments. Thermal treatments were applied in an ultrasonic bath (600 W, 25 kHz, model GAT600W, ATU, Spain) using water as heating fluid.

b) Conventional thermal treatments (CV) where samples were kept in a water bath for 5 h at 50 °C.

2.2. Instrumental adhesiveness

Textural analysis was performed using a texture analyzer (Stable Micro Systems, TA-XT Plus, London, UK) by carrying out a separation test using different load cells with a specific probe. Instrumental adhesiveness was measured in sliced ham samples (1 mm) by applying probe tests and calculating the negative area of a force-time curve in tension tests with a single cycle. The texturometer was equipped with a probe connected to a special device that enables horizontal probe displacement. After the separation of the slices, the probe returned to the initial position. The conditions for the instrumental measurement of adhesiveness of dry cured ham slices were reported by López-Pedrouso et al. (2018). From the graph force *vs*. distance obtained, the adhesiveness was calculated. All the measurements were made in triplicate and carried out at room temperature.

2.3. Moisture content

Moisture content was quantified according to the ISO recommended standards 1442:1997 (ISO, 1997).

2.4. Free Amino acid analysis

The free amino acids were extracted following the procedure described by Lorenzo, Cittadini, Bermúdez, Munekata, and Domínguez (2015b). Amino acids were derivatizated with 6-aminoquinolyl-Nhydroxysuccinimidyl carbamate (Waters AccQ-Fluor reagent kit) and analyzed by RP-HPLC using a Waters 2695 Separations Module with a Waters 2475 Multi Fluorescence Detector, equipped with a Waters AccQ-Tag amino acid analysis column. The results were expressed as mg of free amino acid/100 g of dry matter.

2.5. Volatile compound analysis

The extraction of the volatile compounds was performed using solid-phase microextraction (SPME). A SPME device (Supelco, Bellefonte, USA) containing a fused silica fibre (10 mm length) coated with a 50/30 layer of divinylbenzene/ carboxen/polydimethylsiloxane was used. Chromatographic analyses were carried out under the conditions described by Domínguez, Gómez, Fonseca, and Lorenzo (2014) with modifications, and a gas chromatograph 7890B (Agilent Technologies, Santa Clara, CA, USA) equipped with a mass selective detector 5977B (Agilent Technologies) was used. For extraction, 1 g of each sample was weighed in a 20 mL vial, after being ground using a commercial grinder. The conditioning, extraction and injection of the samples were carried out with an autosampler PAL-RTC 120. Volatile compounds were identified by comparing their mass spectra with those contained in the NIST14 (National Institute of Standards and Technology, Gaithersburg) library, and/or by comparing their mass spectra and retention time with authentic standards (pentane, octane, decane, undecane, dodecane, tridecane, propanal, butanal, pentanal, hexanal, heptanal, octanal, decanal, nonanal and pentadecanal) (Supelco, Bellefonte, PA, USA), and/or by calculation of retention index relative to a series of standard alkanes (C_5–C_{14}) (for calculating Kovats indexes,