



Effects of high-pressure processing on the volatile compounds of sliced cooked pork shoulder during refrigerated storage

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ABSTRACT

The effect of high-pressure processing (pressure levels of 400, 500 and 600 MPa, and exposure times of 5 and 10 min) on the volatile profile of vacuum-packaged sliced cooked pork shoulder held for 28 days at 4 °C was assessed. The volatile fraction of pressurized samples scarcely changed immediately after treatment and remained stable for 14 days, regardless the pressure and time of exposure. After 21 days of storage, significant differences were observed in the profile of volatile compounds in pressurized samples as compared with control samples, these differences being treatment dependent. At the end of the storage period, control and 400 MPa samples showed higher levels of acetic and fatty acids, ethanol and ethyl esters, whereas 500 and 600 MPa samples contained higher levels of ethanal, branched-chain aldehydes, diacetyl, acetoin, and 2,3-butanediol among other compounds. These results suggest that the high-pressure treatment had a discriminant effect on the microbiota of cooked pork shoulder, which led to the accumulation of different volatile compounds during the refrigerated storage of control and pressurized samples.

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1. Introduction

Aroma is a main sensory characteristic of foods. Aroma alterations may become crucial, regarding consumer acceptability, in foods with delicate odour and mild flavour, as is the case of cooked ham or cooked pork shoulder. The aroma of these products is mainly the result of thermally induced reactions, that is, Maillard reaction and lipid degradation (Mottram, 1998). It depends on the temperature and time of processing, but also on factors such as the composition and quantity of the brine injected or the spices added (Schmidt, 1988). Bacterial growth in these products usually results in acidity and formation of off-flavours (Leroy, Vasilopoulos, Van Hemelryck, Falcony, & De Vuyst, 2009; Samelis, Kakouri, Georgiadou, & Metaxopoulos, 1998).

Volatile compounds released from foods are closely related to their aroma. Monitoring the changes in the volatile fraction can help in determining microbial spoilage or possible detrimental effects of technological treatments or prolonged storage periods. Some studies have been performed on the volatile fraction of cooked ham (Chiesa, Soncin, Biondi, Cattaneo, & Cantoni, 2006) and spoiled cooked ham (Leroy et al., 2009). However, scarce information is available on the effect of non-thermal technologies, specifically high-pressure processing (HPP), on the volatile profile of cooked meat products. In previous studies, the differences in the

volatile profile of cooked HPP-treated chicken and beef meat observed with respect to non-pressurized samples, were interpreted as coming from the alteration of the microflora by the action of pressure (Rivas-Cañedo, Fernández-García, & Nuñez, 2009; Rivas-Cañedo, Juez-Ojeda, Nuñez, & Fernández-García, 2010).

In the present study, the evolution of the volatile fraction of pressurized and control sliced cooked pork shoulder was investigated throughout a 28-day vacuum-packaged storage period at 4 °C. Microbiological, physicochemical and sensory characteristics of the product were also monitored.

2. Materials and methods

2.1. Cooked pork shoulders

Five entire vacuum-packed cooked pork shoulders (approximately 3.5 kg in weight) from the same batch were acquired at a manufacturing plant (Liberto de Pedro, S.L., Madrid, Spain). The manufacture procedure is as follows: deboned shoulders are mechanically brine-injected, vacuum-tumbled during 45 min and allowed to cure for 18 h at 12–14 °C. Brine (La Pilarica, Valencia, Spain) consists of a mixture of salt, dextrose, maltodextrin, antioxidants (sodium ascorbate, E-301, and sodium citrate, E-331), emulsifier (sodium polyphosphate, E-452i), gelling agent (carrageenan, E-407), preservative (sodium nitrite, E-250) and aroma. After curing, shoulders are vacuum-packaged in a plastic film and cooked at 80 °C for 3.5 h, reaching an inner temperature of approximately

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Table 1

Effect of pressure level on the microbial counts, pH values and sensory quality of sliced cooked pork shoulder during refrigerated storage (4 °C).

| Days of storage | Pressure level | Microbial counts (log CFU/g) | | pH | Sensory attributes | |
|-----------------|----------------|------------------------------|--------------------------|--------------------------|---------------------------|---------------------------|
| | | MAB ¹ | LAB ² | | Odour quality | Sourness |
| 0 | Untreated | 6.24 ± 1.23 ^a | 6.30 ± 1.06 ^a | 6.55 ± 0.08 ^b | 5.88 ± 0.39 ^a | 0.00 ± 0.00 ^a |
| | 400 MPa | 4.24 ± 0.42 ^b | 2.40 ± 0.53 ^b | 6.50 ± 0.09 ^b | 5.40 ± 0.88 ^{ab} | 0.03 ± 0.08 ^a |
| | 500 MPa | 2.54 ± 0.43 ^c | 1.00 ± 0.76 ^c | 6.53 ± 0.05 ^b | 5.13 ± 0.90 ^b | 0.20 ± 0.39 ^a |
| | 600 MPa | 2.09 ± 0.09 ^d | 1.35 ± 0.37 ^c | 6.63 ± 0.14 ^a | 5.63 ± 0.75 ^{ab} | 0.07 ± 0.16 ^a |
| 14 | Untreated | 8.11 ± 0.01 ^a | 7.83 ± 0.02 ^a | 5.86 ± 0.14 ^b | 4.49 ± 0.74 ^a | 0.23 ± 0.39 ^a |
| | 400 MPa | 6.59 ± 0.11 ^b | 6.18 ± 0.03 ^b | 6.46 ± 0.08 ^a | 4.60 ± 0.36 ^a | 0.02 ± 0.06 ^{ab} |
| | 500 MPa | 6.00 ± 0.22 ^c | 5.83 ± 0.43 ^c | 6.51 ± 0.10 ^a | 4.89 ± 0.48 ^a | 0.00 ± 0.00 ^b |
| | 600 MPa | 5.10 ± 0.36 ^d | 5.16 ± 0.50 ^d | 6.52 ± 0.07 ^a | 4.86 ± 0.69 ^a | 0.03 ± 0.10 ^{ab} |
| 28 | Untreated | 8.78 ± 0.05 ^a | 8.85 ± 0.07 ^a | 5.71 ± 0.12 ^c | 3.89 ± 0.64 ^b | 1.24 ± 0.95 ^a |
| | 400 MPa | 8.41 ± 0.13 ^b | 8.57 ± 0.22 ^b | 5.78 ± 0.07 ^c | 3.88 ± 0.95 ^b | 1.31 ± 1.84 ^a |
| | 500 MPa | 8.39 ± 0.12 ^b | 8.42 ± 0.17 ^c | 6.17 ± 0.15 ^b | 3.97 ± 0.74 ^b | 0.03 ± 0.10 ^a |
| | 600 MPa | 7.72 ± 0.05 ^c | 7.81 ± 0.11 ^d | 6.27 ± 0.08 ^a | 4.89 ± 0.55 ^a | 0.03 ± 0.10 ^a |

¹ MAB, Mesophilic aerobic bacteria.² LAB, Lactic acid bacteria.abcd Mean values (pooled data from 5 min and 10 min treatments) followed by the same letter do not differ significantly ($P < 0.05$) between the different pressure levels within the same week of storage.

67 °C. The plastic film is removed after cooling in order to eliminate the drip loss and the shoulders are vacuum-packaged again and held at 4 °C until marketed. One shoulder was used for the microbiological analysis, a second one for the sensory analysis and the three remaining shoulders were used for the determination of both pH and volatile compounds.

2.2. High pressure treatments

At the laboratory, the plastic film was taken off, both ends of each shoulder were rejected and the rest was cut into 4 mm-thick slices. Each slice was wrapped in aluminium foil and vacuum-packaged in two multilayer plastic bags (HT 3050, Cryovac Sealed Air Corporation, Milano, Italy) resistant to HPP. The slices from each shoulder were divided into seven sets, six of which were subjected to different HPP-treatments while one was kept untreated as control.

Six pressure–time combinations were applied, using three pressure levels (400, 500 and 600 MPa) and two times of exposure (5 and 10 min). HPP-treatments were carried out at 12 °C in a 100 L capacity discontinuous isostatic press at NC Hyperbaric (Burgos, Spain). Come-up times were 79, 98 and 120 s for pressures of 400, 500 and 600 MPa, respectively, while come-down times were less than 5 s. After pressurization, cooked pork shoulder slices were stored at 4 °C for up to 28 days. Analyses were carried out after 0, 7, 14, 21 and 28 days of storage.

Volatile compounds and pH were determined in triplicate in such a way that one slice per individual shoulder (3) and per storage time (5) was kept as control and another slice, also per shoulder and per storage time, was subjected to one of the six high pressure treatments. The total number of samples analysed was 7 treatments × 5 storage times × 3 shoulder replicates = 105. Samples were not frozen before analysis in order to avoid artefact formation.

2.3. Microbial analysis and pH

A 10 g representative cooked pork shoulder sample was homogenized with 90 ml of sterile 0.1% (wt/vol) peptone water solution containing 8.5 g/L of sodium chloride in a Colworth Stomacher 400 (A. J. Seward Ltd., London, UK). Mesophilic aerobic bacteria (MAB), lactic acid bacteria (LAB) and Gram-negative bacteria (GNB) were determined on duplicate plates of Tryptic Soy Agar (TSA; Biomedics), M17 agar (Biolife; acidified at pH 5.7 with 5 M acetic acid), and PMK agar (Biolife), respectively, all incubated for

72 h at 30 °C. Enterococci were determined on Kenner Fecal (KF) Agar plates (Scharlau Microbiology, with 1% of 2,3,5-triphenyl-tetrazoliumchlorid, TTC) incubated for 48 h at 37 °C. Yeasts were determined on Chloramphenicol Glucose Agar plates (CGA; Scharlau Microbiology) incubated for 5 days at 22 °C.

The pH was determined with a pH-meter (model GLP 22, Crison Instruments, Barcelona, Spain) on a homogenate obtained by blending 10 g of ground cooked pork shoulder with 20 mL of Milli-Q water with an Ultraturrax (T18 basic, IKA, Staufen, Germany) and allowing to rest for 10 min.

2.4. Sensory analysis

Sensory analysis of cooked pork shoulder was performed at days 0, 7, 14, 21 and 28 of storage by a trained 10-member panel. Panellists were presented coded Petri dishes in randomized order containing small squares (2 × 2 cm) of slices of the HPP-treated and control samples. A recently manufactured shoulder was used as a reference. Panellists were asked to score the odour quality on a 0–7 point scale. Off-odours putrid, sour, alcoholic, fruity and ammonia were also scored on a 0–7 point scale.

2.5. Volatile compound analysis

Volatile compounds were extracted by solid-phase microextraction (SPME) and analysed by gas chromatography-mass spectrometry (GC-MS) (HP 6890-MSD HP 5973, Agilent, Palo Alto, CA). A cooked pork shoulder sample (15 g) was homogenized in a mechanical grinder with 22.5 g of anhydrous sodium sulphate (Na₂SO₄) and 30 µL of an aqueous solution of 250 mg/L camphor added as internal standard. A 40 mL headspace glass vial was filled with the mixture (12.00 ± 0.01 g), sealed with a PTFE (polytetrafluoroethylene) faced silicone septum and submerged in a thermostatic bath at 40 °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 fibre (Supelco, Bellefonte, PA) was inserted through the PTFE septum for headspace extraction, after which it was inserted into the GC injection port for desorption (260 °C/10 min in splitless mode). Before use, the fibre was conditioned in the GC injection port at 270 °C for 1 h as recommended by the manufacturer. Two fibres were used for the whole study.

Chromatographic separation was carried out in a Zebron 100% polyethylene glycol capillary column (60 m long; 0.25 mm internal

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