Influence of high-pressure processing at different temperatures on free amino acid and volatile compound profiles of dry-cured ham

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Abstract

The effect of high pressure processing (HPP) (600 MPa during 6 min) at different temperatures (0, 20 and 35 ºC) in dry-cured ham has been studied in order to optimize the technique and reduce its impact on chemical characteristics, which are widely related with sensorial parameters. Vacuum-packed slices from 120 dry-cured hams were used. These slices were submitted to four different treatments: without application of pressure or temperature (CO), high pressure treatment at 0° C (HPP-0), high pressure treatment at 20° C (HPP-20), and high-pressure treatment at 35 °C (HPP-35). The effect of the treatments on free amino acids and volatile compounds profile was evaluated. The HPP-35 treatment significantly ($P<0.001$) increased the total free amino acid content (6415.63 mg/100 g dry matter) when compared to the contents of the CO, HPP-0 and HPP-20 treatments (5313.16, 4787.30 and 5072.48 mg/100 g dry matter, respectively). Significant differences were also found among treatments in the content of 13 individual free amino acids, and HPP-35 samples presented the highest values in 12 of them. Similarly, the total volatile compound content was influenced by temperature-assisted HPP treatments. The HPP-35 treated samples showed the highest content (78415.27 AU x 10^3/g dry-cured ham) and the HPP-0 treated samples the lowest content (28584.14 AU x 10^3/g dry-cured ham). No significant differences were observed between CO and HPP-20 treatments. The fractions of volatile compounds derived from lipolysis, proteolysis and microbial activity were significantly modified by the different treatments. HPP-0 samples presented lower values of alcohol and hydrocarbon contents, whereas HPP-35 samples showed higher ketone and ester contents.

Keywords: High hydrostatic pressure, volatile compound, free amino acids, dry-cured ham

1. Introduction

The use of high pressure processing (HPP) in food technology began in the 90s. Since then, most of the studies have focused on reducing the microbial load of food and obtaining safer products with a longer shelf life. According to Duranton *et al.* (2014), HPP has other potential applications. For instance, HPP has been studied as an auxiliary method in the food elaboration processes in recent years. In this line of thought, Duranton *et al.* (2012) used HPP on salting stage of dry-cured ham processing to improve salt diffusion and to reduce the amount of salt in the formulation. However, some studies found that, under certain conditions, HPP can cause physico-chemical, sensory, and even functional alterations, particularly on proteins, lipids and starches (Rivalain, Roquain, and Demazeau 2010; Liu, Selomulyo, and Zhou 2008). Since some protein conformations are sensitive to pressure, the application of high pressure treatments can induce modifications on enzyme activity (Chéret *et al.*, 2006; Buckow, Quong, and Versteeg 2010), which can result in texture changes, mainly by increasing hardness and elasticity of the product (Yoshioka and Yamada, 2002; Duranton *et al.*, 2012). It is worth mentioning that Tao, Sun, Hogan and Kelly (2014) concluded that moderate pressure does not cause significant changes in the flavor of products when high hydrostatic pressures were used for sterilization.

The most important attributes affecting consumer´s purchase preference are related to odour and taste. The aroma is originated by chemical and enzymatic reactions during the processing of dry-cure hams (Bermúdez *et al.* 2015). Concerning the effect of HPP on the enzymes that originate flavor compounds, contrasting results have been reported in scientific literature. In this way, both Clariana *et al.* (2011) and Clariana *et al.* (2012) observed an increase in the superoxide dismutase activity, but no effect was shown on catalase and glutathione peroxidase activity after HPP treatments at 400 MPa. Conversely, superoxide dismutase and glutathione peroxidase activities were reduced without any effect on catalase activity after treatment at 900 MPa. Nevertheless, HPP at 600 MPa showed no effect on the activity of none of the antioxidant enzymes.

Moreover, the compositional characteristics of the product could influence the volatile compound profile. For example, high intramuscular fat content in ham can increase the concentration of compounds such as acetic acid, methylbenzene or phenol, whereas low intramuscular fat content can lead to greater contents of 2-propanol and dimethyl sulfide, among others (Martínez-Onandi *et al.* 2016a). On the other hand, the chemical and enzymatic reactions during the process involve the modification of protein structures in order to develop a particular ham taste. Due to the fact that HPP treatment could promote changes in cellular structures (dos Santos Aguilar, Cristianini, & Sato 2018) and temperature could have an impact in the development of reactions, it is emerging the necessity to find out the consequences in the final flavor after the application of this technique.

In this way, interesting results were obtained by using HPP technique during pre and post rigor stage of dry-cured ham to improve texture (Fulladosa *et al.*, 2009) and in vacuum-packaged products to enhance shelf life (Fuentes *et al.* 2010). The impact of HPP on sensory properties of the packaged dry-cured ham was previously studied regarding to the pressure effect, but there are no studies about the combined effect of temperature and HPP processing in volatile and free amino acid composition of dry-cured ham. Due to the multitude of current applications of the HPP as well as their potential uses in the future, it is interesting to study the impact of the HPP on chemical changes as a first step to understand the effects on sensory attributes. Therefore, the objective of this study was to evaluate the effect of HPP treatment assisted with three different temperatures on free amino acid content and volatile compound of dry-cured ham.

2.1. Materials and methods

2.1. Samples

One hundred and twenty raw hams with pH<5.5, which are more prone to develop defective texture properties, from animals belonging to crosses of Large White and Landrace breeds (medium fat content) were obtained from a commercial slaughterhouse. All hams were weighted (11.9 kg $\pm$ 1.1 kg) and manufactured according to the traditional system. Dry-cured hams, the aitch bone, the butt and the femur bone were excised and the cushion part, containing *Biceps femoris* (BF) muscle, was obtained and trimmed.

After that, the 120 hams were divided into treatments (30 hams per treatment). From each ham unit, three 1.5 mm-thick slices were vacuum packed in individual plastic bags of polyamide/polyethylene (oxygen permeability of 50 cm^3/m^2/24h at 23ºC and water permeability of 2.6 g/m^2/24h at 23ºC and 85% RH, Sacoliva® S.L., Spain) and stored in a chamber at 4 ºC $\pm$ 2 °C until the treatment application.

2.2. HPP treatments

The treatment of the packaged slices was applied using a NC Hyperbaric WAVE 6000/120 equipment (NC Hyperbaric, Burgos, Spain). Three different treatments were performed at 600 MPa during 6 min, each one accompanied by a different temperature: the first at 0 °C (HPP-0), the second at 20 °C (HPP-20) and the third at 35 °C (HPP-35). In order to evaluate the effects of HPP treatments, a fourth group of samples was not treated and was used as a control (CO) batch.

2.3. Free amino acid analysis

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

2.4. Volatile compound analysis

For the volatile compound extraction, a solid-phase micro extraction (SPME) device (Supelco, Bellefonte, PA, USA) containing a fused-silica fibre (10 mm length) coated with a 50/30 mm thickness of DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethylsiloxane) was used. For the volatile compound determination, a gas chromatograph 7890B (Agilent Technologies, Santa Clara, CA, USA) equipped with a DB-624 capillary column (30 m, 0.25 mm i.d., 1.4 μm film thickness; J&W Scientific, Folsom, CA, USA) coupled to a mass selective detector 5977B (Agilent Technologies) was used.

The extraction of the volatile compounds (SPME) was performed following the procedure described by Domínguez, Gómez, Fonseca, & Lorenzo (2014) with some modifications. One g of each sample (after being ground using a commercial grinder) was weighed in a 20 mL vial. The vials were subsequently screw-capped with a laminated Teflon-rubber disc. The fibre was previously conditioned by heating in a Fiber Conditioning Station at 270 °C for 30 min. The conditioning, extraction and injection of the samples were carried out with an autosampler PAL-RTC 120. The extractions were carried out at 37 °C for 30 min, after equilibration of the samples for 15 min at the temperature used for extraction, which ensured a homogeneous temperature for both sample and headspace. Once sampling was finished, the fibre was transferred to the injection port of the gas chromatograph–mass spectrometer (GC–MS) system. The SPME fibre was desorbed and maintained in the injection port at 260 °C during 8 min. The samples were injected

134 in splitless mode. Helium was used as a carrier gas with a flow of 1.2 mL/min (9.59 psi). The
135 temperature program was firstly isothermal for 10 min at 40 °C, then raised to 200 °C at 5 °C/min
136 and next to 250 °C at 20 °C/min, and finally held for 5 min; total run time was 49.5 min. Injector
137 and detector temperatures were both set at 260 °C. The mass spectra were obtained using a mass
138 selective detector working in electronic impact at 70 eV, with a multiplier voltage of 850 V and
139 collecting data at 6.34 scans/s over the range m/z 40–550. Compounds were identified by
140 comparing their mass spectra with those contained in the NIST14 (National Institute of Standards
141 and Technology, Gaithersburg) library, and/or by comparing their mass spectra and retention time
142 with authentic standards (Supelco, Bellefonte, PA, USA), and/or by calculation of retention index
143 relative to a series of standard alkanes (C5–C14) (for calculating Kovats indexes, Supelco 44585-
144 U, Bellefonte, PA, USA) and matching them with data reported in literature. The results were
145 expressed as quantified area units (AU) $\times 10^3$/g of sample.

146 *2.5. Statistical analysis*

147 The effect of treatments was examined using a one-way ANOVA. When a significant effect
148 ($P<0.05$) was detected, means were compared using Tukey´s test. Analyses were conducted using
149 the IBM SPSS Statistics 19.0 (IBM Corporation, Somers, NY, USA) software package.

150 **3. Results and discussion**

151 *3.1. Free amino acids*

152 Table 1 shows the effect of different HPP-temperature treatments on the free amino acid
153 content (expressed as mg/100 g dry matter) of dry-cured ham. Statistical analysis showed that
154 total free amino acid content was significantly ($P<0.001$) affected by treatments. HPP-35 group
155 displayed the highest values (5313.16 *vs*. 4787.30 *vs*. 5072.48 *vs*. 6415.63 mg/100 g dry matter
156 for CO, HPP-0, HPP-20 and HPP-35 tretaments, respectively). No significant differences were
157 observed among CO, HPP-0 and HPP-20 treatments. These values were in the range values 4000-
158 7000 mg/100g dry matter that was reported in previous studies (Bermúdez, Franco, Carballo,
159 Sentandreu, & Lorenzo, 2014; Pérez-Santaescolástica *et al.* 2018a; Pérez-Santaescolástica *et al.*
160 2018b) about dry-cured ham volatile composition. The higher total free amino acid content in
161 HPP-35 samples was expected since it is well known that proteins are greatly influenced by
162 temperature, so their structures could be degraded into smaller amino acids. In this regard, 13 of
163 the 18 amino acids studied were significantly influenced by temperature-assisted HPP treatments.
164 The samples submitted to HPP at 35 °C had the highest content in 12 amino acids (aspartic acid,
165 serine, glutamine, glycine, histidine, taurine, arginine, threonine, alanine, cysteine, valine, and
166 lysine). Tyrosine was the only amino acid that presented the highest level in untreated samples.

167 Changes in individual amino acid content could promote changes in the final flavor of dry-
168 cured ham (Jurado *et al.*, 2007; Hidalgo & Zamora, 2004). Thereby, the higher content in specific
169 amino acids showed in HPP-35 samples may influence the perception of sweet (calculating as
170 sum of alanine, serine, proline, threonine and glycine content), acid (calculating as sum of
171 phenylalanine, histidine, glutamic and aspartic acid content) and aged (calculating as sum of
172 lysine, tyrosine and aspartic acid content) attributes in comparison to other treated and untreated
173 samples (Table 1). In addition, previous studies showed that an increment of bitter taste in hams
174 could be attributed to excessive proteolysis (Careri *et al.*, 1993; Parolari, Virgili, & Schivazappa,
175 1994). However, the amino acids responsible for the bitter taste were not affected by any treatment
176 in the present study.

177 *3.2. Volatile compounds*

Significant differences ($P<0.001$) among treatments were found in the total content of volatile compounds. The highest values were observed in the HPP-35 batch (78415.27 AU x 10^3/g of dry-cured ham) while the lowest contents were obtained from the HPP-0 batch (28584.14 AU x 10^3/g of dry-cured ham) (Table 2). In comparison to HPP-0 treatment, the samples showed significant declines in hydrocarbons, aldehydes, alcohols, carboxylic acids, sulphur compounds and chloro compounds content by 55%, 56%, 40%, 69%, 85% and 65%, respectively. Aldehydes, alcohols, carboxylic acids, nitrogenous and sulphur compounds content were reduced by 44%, 18%, 34%, 28% and 91%, respectively, in HPP-20 treated samples, while hydrocarbons, ketones and chloro compounds were incremented by 60% 58% and 79%, respectively, in comparison to CO. Furthermore, samples treated with HPP at 35 °C presented reduction in the aldehydes, carboxylic acid and sulphur compounds (22%, 36% and 82%, respectively) while hydrocarbons, ketones, ether and esters and chloro compounds were incremented by 109%, 109%, 37% and 69%, respectively, in comparison to CO. It is well known that aldehydes, ketones, ester and ethers, and alcohols (to a limited extent) are the main families associated with the aroma of dry-cured ham (Carrapiso *et al.*, 2010; García-González *et al.*, 2008). Therefore the temperature-assisted HPP treatments may affect the quality of the final product. In this way, the HPP-35 treatment enhanced ester and ether contents, which are responsible for fruity odour notes. Meanwhile, all of HPP treatments caused a significant reduction of sulphur compounds, a fact that could modify the aroma by incrementing rotten egg and burnt notes. In addition, our data are in agreement with the results obtained by Martínez-Onandi *et al.* (2016b) in sliced Serrano dry-cured ham treated at 600 MPa and 21 °C for 2.5 min.

A total of 149 volatile compounds were identified and classified based on their origin according to Narváez-Rivas *et al.* (2012), Martín *et al.* (2006) and Fonseca *et al.* (2015). Of the 149 compounds, 92 were presumably originated from lipid oxidation, 21 were derived from proteolysis reactions, 21 were attributed to microbial activity and 15 had an unknown origin. Table 3 lists the compounds detected in the volatile fraction of the slices of dry-cured ham, as well as the effect of HPP treatments, the linear retention indexes, the ions used for quantification and the method used for identification.

147 out of the 149 identified volatile compounds were significantly influenced by the HPP treatment. Regarding the origin of these compounds, the most probable origin was lipolysis, followed by proteolysis and microbial activity. The sum of secondary products of lipid oxidative decomposition was around 80% of the total volatile content in all treatments with the exception of HPP-0, in which such compounds accounted for 67%. In contrast, the compounds derived from proteolysis represented 20% of the total volatile compounds in the HPP-0 group and 8-9% in the other treatments. These differences within families can be explained by the high temperatures (which promote lipolysis) and HPP that can induce protein denaturation (Guyon *et al.* 2018). Similar results were found by Martínez-Onandi *et al.* (2017) and Ramírez and Cava (2007) who reported values around 75% and 81.6% of total compounds were associated with lipid oxidation, respectively, and values of 20% and 12.7% of total compounds were attributed to proteolysis, respectively. Previous studies observed that the application of HPP at pressures below 300 MPa have minimum effect on lipid oxidation but higher pressures give an increase in the amount of aldehydes derived from lipolysis (Andrés *et al.*, 2004; Fuentes *et al.*, 2010). In contrast, Martínez-Onandi *et al.* (2016a) did not find any significant effect on linear aldehydes content in dry-cured ham treated at 600 MPa, and these authors concluded that HPP only influenced volatile compounds originated from microbial activity. Moreover, the majority of the most abundant volatile compounds were obtained in either CO or HPP-35 samples (64 and 61 compounds, respectively).

225   Among the lipolysis-derived compounds, hexanal was the most abundant, particularly in
226   untreated samples. Conversely, the lowest value was observed in the HPP-0 batch. Interestingly,
227   an increasing trend in hexanal content was observed as the temperature of treatment increased.
228   This fact can be explained by the potential protective effect of the HPP against hexanal generation.
229   This finding could be considered positive since high levels of this compound gives rancid notes
230   to ham. In contrast, the aroma can turn grassier and more pleasant because of the hexanal
231   reduction (Aparicio & Morales, 1998). On the contrary, previous studies about the effects of HPP
232   on dry-cured hams showed that HPP increased the rancid odor perception due to an increment in
233   aldehydes (Fuentes *et al.*, 2010; Clariana *et al.*, 2011). In agreement to Martinez-Onandi et al
234   (2017), nonanal, propinoic acid, butanoic acid, pentanoic acid, hexanoic acid and pentanal showed
235   higher levels in untreated than in HPP samples. However, lower values of 2-pentanol were
236   obtained from untreated samples (Table 3). Additionally, 1-Octen-3-ol, a characteristic compound
237   of dry-cured ham with a very low threshold in "Montanera hams" (Jurado *et al.*, 2009), did not
238   show significant differences between CO and HPP-35 samples, although the other two treatments
239   (HPP-0 and HPP-20) showed significant lower values.

240   As expected, the main microbial activity-derived compounds detected in this study were
241   esters whose formation are closely related to microbial activity (Ramírez and Cava, 2007). Also,
242   it is well known that temperature affects the ester compounds formation (Gorvatov &
243   Lyaskovkaya, 1980). For this reason, it was no strange that CO and HPP-35 samples presented
244   higher amounts of microbial activity-derived compounds than HPP-0 and HPP-20 samples, and,
245   in the same way, the HPP-20 group presented higher values than HPP-0. Dimethyl disulfide was
246   the main compound detected in CO samples, but it was greatly reduced by HPP treatments
247   (1786.20 AU x 10^3/g of dry-cured ham *vs* 160.12 AU x 10^3/g of dry-cured ham *vs* 61.83 AU x
248   10^3/g of dry-cured ham *vs* 213.32 AU x 10^3/g of dry-cured ham for CO, HPP-0, HPP-20 and HPP-
249   35, respectively). Although the origin of dimethyl disulfide is usually related to the microbial
250   activity, some previous studies established that amino acid catabolism can be another possible via
251   (Sabio *et al.*, 1998; Ramírez and Cava, 2007). Moreover, Muriel *et al.* (2004) found that dimethyl
252   disulfide could result from the reaction between lipid oxidation products and cysteine. In the
253   present study, a positive and significant ($P<0.05$) correlation between cysteine and dimethyl
254   disulfide (r=0.200) was observed and therefore this via for the dimethyl disulfide formation can
255   not be discarded.

256   The compounds derived from proteolysis found in the present study that have been
257   previously detected in dry-cured ham were 2-methyl propanal, 3-methyl butanal, 2-methyl butanal
258   and 2-methyl-2-butenal (Timón *et al.*, 2001; Andrés *et al.*, 2002; Sánchez-Peña *et al.*, 2005). The
259   highest values of 2-methyl propanal, 3-methyl butanal and 2-methyl-2-butenal were observed in
260   HPP-35 samples. Particularly for 2-methyl butanal, all HPP-treated samples (independently of
261   assisted temperature) displayed higher values than CO samples, which may be due to the HPP
262   effect on protein structures. Moreover, the statistical analysis showed a positive correlation
263   (r=0.263, $P<0.01$) between 2-methyl butanal and isoleucine. The degradation of isoleucine is the
264   most probable origin of this compound, as reported by previous studies (Ramírez and Cava, 2007).

265   Finally, fifteen compounds were classified as "unknown origin" whose probable
266   via/reaction was not found in literature. Since their origin is not clear, it is not possible to include
267   them into the three principal treatments already commented. It is worth mentioning that the
268   presence of *p*-Cresol could be associated with animal feed and further accumulation in the animal
269   tissues (Sánchez-Peña *et al.*, 2005; Sabio *et al.*, 1998). The HPP-35 and CO samples showed
270   higher contents of *p*-Cresol than HPP-0 and HPP-20 samples. Aromatic and ciclyc hidrocarbons

were also found: 1,3-dimethyl benzene, 1-ethyl-3-methyl cyclopentane and ethyl cyclopentane contents were reduced by HPP treatment.

4. Conclusion

HPP is a promising technology to process food, specially products affected by higher temperatures. From the results obtained in the present study, it can be concluded that HPP can be applied to dry-cured ham but in the range 0-20 °C in order to minimize the impact of such treatments on free amino acid and volatile compounds. This recommendation is supported by the intense modifications caused HPP and high temperature (particularly at 35 °C) on free amino acid profile and volatile composition, which could reduce product quality.

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Table 1. Effect of different HPP treatments on free amino acids content (expressed as mg/100 g dry matter) of dry-cured ham. Values are means of thirty hams for each treatment.

	Treatment				SEM	*p-value*
	CO	HPP-0	HPP-20	HPP-35		
Aspartic acid	185.15[b]	119.45[a]	145.99[a]	240.20[c]	5.643	<0.001
Serine	201.65[a]	198.30[a]	200.45[a]	251.15[b]	5.647	0.001
Glutamine	450.17[a]	382.41[a]	424.71[a]	588.97[b]	12.112	<0.001
Glycine	196.45[a]	202.46[a]	200.03[a]	238.69[b]	4.444	0.001
Histidine	102.51[b]	82.62[a]	87.44[ab]	127.69[c]	2.879	<0.001
Taurine	93.22[ab]	83.48[a]	92.04[ab]	102.21[b]	2.305	0.045
Arginine	410.98[b]	295.84[a]	346.32[ab]	513.00[c]	11.986	<0.001
Threonine	221.57[ab]	223.76[ab]	216.03[a]	254.45[b]	5.278	0.037
Alanine	419.87[a]	471.30[a]	478.99[ab]	546.58[b]	10.754	<0.001
Proline	287.50	276.28	286.14	314.32	5.691	0.105
Cysteine	346.79[b]	61.44[a]	51.44[a]	553.79[c]	23.108	<0.001
Tyrosine	202.96[c]	116.37[a]	160.34[b]	121.24[a]	4.777	<0.001
Valine	393.22[a]	461.58[b]	471.45[b]	441.94[ab]	8.847	0.007
Methionine	203.84	205.41	216.23	223.19	4.303	0.330
Lysine	265.09[a]	256.41[a]	290.14[a]	448.34[b]	10.239	<0.001
Isoleucine	351.76	377.63	387.34	403.47	7.937	0.119
Leucine	588.53	629.06	654.06	664.31	12.864	0.146
Phenylalanine	391.88	343.50	363.34	382.06	7.045	0.086
TOTAL	**5313.16[a]**	**4787.30[a]**	**5072.48[a]**	**6415.63[b]**	**112.28**	**<0.001**
Sweet[1]	1310.60[a]	1372.10[a]	1379.22[a]	1587.65[b]	29.320	0.003
Bitter[2]	1921.99	2017.17	2092.42	2083.20	38.731	0.362
Acid[3]	737.84[b]	605.81[a]	667.62[ab]	956.87[c]	20.453	<0.001
Aged[4]	653.20[b]	513.87[a]	601.86[ab]	833.88[c]	17.725	<0.001

[a-c] Mean values in the same row (corresponding to the same amino acid/sensory attribute) not followed by a common letter differ significantly ($P < 0.05$; Tukey´s Test)

SEM: standard error of mean.

Treatments: CO= control (without treatment); HPP-0=High pressure treatment at 0 ºC; HPP-20=High pressure treatment at 20 ºC; HPP-35=High pressure treatment at 35 ºC

[1]Sweet flavor = $\sum$ of alanine, glycine, threonine, serine and proline; [2]Bitter flavor = $\sum$ of leucine, valine, isoleucine, methionine and phenylalanine; [3]Acid flavor = $\sum$ of glutamic acid, aspartic acid and histidine; [4]Aged flavor = $\sum$ of lysine, tyrosine and aspartic acid

450 **Table 2.** Levels (expressed as quantified area units (AU) x 10^3/g dry cured ham) of the mainly families
451 of volatile compounds identified in untreated and HPP at 0° C, 20° C and 35° C treated dry cured ham.
452 Values are means of thirty hams for each treatment.

Compound	Treatment				SEM	*p-value*
	CO	**HPP-0**	**HPP-20**	**HPP-35**		
Aliphatic hydrocarbons	20538.44[b]	9197.34[a]	33631.08[c]	43688.86[d]	1469.501	<0.001
Aromatic and cyclic hydrocarbons	902.09[c]	486.35[a]	747.07[b]	1027.21[c]	27.306	<0.001
Hydrocarbons	21440.53[b]	9683.69[a]	34385.34[c]	44716.06[d]	1489.524	<0.001
Aldehyde	22467.49[c]	9840.67[a]	12562.24[a]	17443.40[b]	607.511	<0.001
Ketone	2454.69[a]	2166.36[a]	3890.57[b]	5138.82[c]	124.636	<0.001
Esther and ether	1659.26[a]	1608.06[a]	1761.60[a]	2272.76[b]	43.336	<0.001
Alcohol	6465.70[c]	3900.03[a]	5295.68[b]	6826.08[c]	161.750	<0.001
Carboxylic acid	1027.64[c]	321.09[a]	680.18[b]	660.01[b]	31.479	<0.001
Nitrogenous compounds	585.65[bc]	524.52[b]	422.16[a]	648.02[c]	13.799	<0.001
Sulphur compounds	2178.20[b]	321.41[a]	206.63[a]	400.44[a]	88.244	<0.001
Chloro compounds	270.11[b]	121.20[a]	482.85[c]	455.68[c]	16.934	<0.001
Total Compounds	**58549.27[b]**	**28487.02[a]**	**59641.13[b]**	**78561.29[c]**	**1986.982**	**<0.001**

453 [a-d] Mean values in the same row (corresponding to the same family) not followed by a common letter differ
454 significantly ($P<0.05$; Tukey´s Test).

455 SEM: standard error of mean. Treatments: CO= control (without treatment); HPP-0=High pressure treatment at 0 ºC;
456 HPP-20=High pressure treatment at 20 ºC; HPP-35=High pressure treatment at 35 ºC.

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Table 3. Effect of treatments on volatile compound content (expressed as quantified area units (AU) x 10^3/g dry cured ham). Values are means of thirty hams for each treatment.

Compound	m/z	LRI	R	CO	HPP-0	HPP-20	HPP-35	SEM	p-value
					Treatment				
Pentane [Y]	43	516	*ms, lri, s*	1166.82[c]	341.36[a]	568.52[b]	722.43[b]	39.322	<0.001
Propanal [Φ]	58	526	*ms, lri, s*	133.06[b]	26.37[a]	38.29[a]	42.57[a]	5.109	<0.001
Acetone [Φ]	58	528	*ms, lri*	171.04[ab]	256.36[b]	151.17[a]	387.56[c]	14.734	<0.001
Isopropyl Alcohol [YΦ]	45	532	*ms, lri*	88.41[a]	218.13[c]	191.73[b]	226.23[c]	6.570	<0.001
2,3-Hexanedione [Φ]	41	562	*ms, lri*	356.04[a]	290.13[a]	1252.05[b]	2096.88[c]	84.736	<0.001
n-Hexane [Y]	57	562	*ms, lri, s*	822.88[a]	706.36[a]	2744.00[b]	4705.52[c]	185.161	<0.001
1-Butene, 2,3-dimethyl- [Y]	69	571	*ms, lri*	12.15[a]	22.71[b]	14.63[a]	30.09[c]	1.043	<0.001
1-Propanol [YΦ]	59	572	*ms, lri*	27.91[a]	42.80[b]	52.10[bc]	57.38[c]	1.885	<0.001
Butanal [YΦ]	72	584	*ms, lri, s*	24.28[c]	8.00[a]	11.96[ab]	15.52[b]	0.808	<0.001
2-Butanone [Φ]	72	596	*ms, lri*	182.67[a]	255.04[b]	281.36[b]	247.02[b]	11.320	0.012
2-Butanol [YΦ]	45	607	*ms, lri*	13.92[a]	28.77[bc]	24.25[b]	33.51[c]	1.009	<0.001
Cyclopentanone, 3-methyl- [Y]	56	667	*ms, lri*	45.68[c]	9.53[a]	16.76[a]	28.13[b]	1.683	<0.001
Heptane [Y]	71	675	*ms, lri, s*	1321.57[c]	203.20[a]	353.71[ab]	555.71[b]	49.078	<0.001
Furan, 2-ethyl- [YΦ]	81	703	*ms, lri*	40.15[c]	7.25[a]	12.78[ab]	16.35[b]	1.492	<0.001
1-Butanol [YΦ]	56	707	*ms, lri*	17.51[a]	16.42[a]	27.14[b]	32.09[b]	1.027	<0.001
2-Pentanone [Φ]	86	720	*ms, lri*	98.54[b]	59.43[a]	86.95[ab]	144.70[c]	4.956	<0.001
Pentanal [YΦ]	57	728	*ms, lri, s*	1190.56[c]	378.49[a]	491.34[a]	771.43[b]	44.792	<0.001
1-Penten-3-ol [YΦ]	57	730	*ms, lri*	1099.81[c]	350.04[a]	552.91[b]	666.28[b]	35.716	<0.001
2-Pentanol [YΦ]	45	751	*ms, lri*	86.03[a]	311.24[b]	222.00[b]	413.17[c]	17.625	<0.001
Pentane, 2,3,4-trimethyl- [Y]	71	756	*ms, lri*	181.85[b]	115.66[a]	98.42[a]	108.51[a]	6.903	<0.001

Compound		LRI	ID					SEM	p
Pentane, 2,3,3-trimethyl-[Y]	71	763	*ms, lri*	252.09[b]	184.67[a]	125.65[a]	131.57[a]	9.770	<0.001
Pentane, 3-ethyl-[Y]	70	770	*ms, lri*	46.85[b]	25.18[a]	15.13[a]	15.93[a]	1.820	<0.001
1-Pentene, 3-ethyl-2-methyl-[Y]	83	774	*ms, lri*	32.62[b]	14.34[a]	55.02[c]	85.19[d]	2.815	<0.001
Hexane, 2,2,5-trimethyl-[Y]	57	800	*ms, lri*	355.20[c]	198.20[b]	86.68[a]	85.66[a]	15.519	<0.001
Octane[Y]	85	822	*ms, lri, s*	3308.36[c]	587.64[a]	907.92[b]	1277.65[b]	124.815	<0.001
Propanoic acid[YΦ]	74	827	*ms, lri*	12.64[c]	3.76[a]	8.35[b]	8.05[b]	0.591	<0.001
2-Octene, (E)-[Y]	112	833	*ms, lri*	342.80[c]	75.77[a]	120.74[a]	208.17[b]	12.002	<0.001
Heptane, 3,4,5-trimethyl-[Y]	85	842	*ms, lri*	76.88[c]	49.25[b]	9.68[a]	9.50[a]	3.621	<0.001
3-Octene, (E)-[Y]	112	845	*ms, lri*	170.74[c]	39.14[a]	55.81[a]	99.32[b]	6.553	<0.001
1-Pentanol[YΦ]	55	847	*ms, lri, s*	500.17[c]	136.34[a]	220.83[a]	385.19[b]	19.128	<0.001
Hexanal[YΦΨ]	56	865	*ms, lri*	15270.28[d]	3980.78[a]	6595.32[b]	9404.24[c]	510.237	<0.001
Hexane, 2,2,5,5-tetramethyl-[Y]	57	914	*ms, lri*	387.10[b]	304.54[b]	87.89[a]	130.89[a]	17.892	<0.001
Butanoic acid[YΦ]	60	918	*ms, lri*	191.58[c]	50.50[a]	113.16[b]	69.28[a]	7.191	<0.001
4-Nonene[Y]	70	926	*ms, lri*	198.55[b]	128.93[a]	152.16[a]	230.92[b]	7.011	<0.001
Heptane, 2-methyl-3-methylene-[Y]	126	930	*ms, lri*	17.87[a]	11.42[a]	17.45[a]	28.24[b]	1.028	<0.001
Nonane[Y]	57	936	*ms, lri, s*	201.88[c]	123.93[b]	57.32[a]	84.50[ab]	7.840	<0.001
2-n-Butyl furan[Y]	81	944	*ms, lri*	39.67[b]	13.34[a]	20.37[a]	39.72[b]	1.702	<0.001
3-Heptanone[Φ]	57	960	*ms, lri*	42.37[a]	47.19[a]	70.34[b]	117.12[c]	3.546	<0.001
2-Heptanone[Y]	58	967	*ms, lri*	455.34[a]	344.09[a]	434.04[a]	620.46[b]	19.443	<0.001
Heptanal[YΦ]	70	974	*ms, lri, s*	988.97[c]	251.92[a]	396.51[ab]	512.02[b]	33.687	<0.001
2-Nonen-4-one[Φ]	69	979	*ms, lri*	16.18[ab]	14.73[a]	15.98[ab]	20.45[b]	0.671	0.014
2-Octene, 4-ethyl-[Y]	69	982	*ms, lri*	146.99[b]	106.14[a]	106.60[a]	126.89[ab]	5.230	0.013
Octane, 3-methyl-6-methylene-[Y]	70	985	*ms, lri*	303.36[bc]	198.09[a]	243.42[ab]	365.97[c]	13.905	<0.001

Compound								SEM	p-value
Octane, 4-ethyl- [Y]	69	991	*ms, lri*	90.08[b]	72.48[ab]	68.06[a]	83.64[ab]	2.594	0.007
2-Hepten-4-one, 6-methyl- [Y]	69	992	*ms, lri*	91.95[ab]	73.46[a]	71.13[a]	102.44[b]	3.405	0.001
Pentane, 3,3-dimethyl- [Y]	85	995	*ms, lri*	9.35[b]	5.33[a]	4.84[a]	7.22[ab]	0.400	<0.001
Methional [YΦ]	104	999	*ms, lri*	201.58[a]	211.98[a]	252.49[a]	387.58[b]	15.839	<0.001
Nonane, 2,3-dimethyl- [Y]	71	1003	*ms, lri*	87.74[c]	62.48[b]	40.21[a]	63.90[b]	3.371	<0.001
1-Octene, 2,6-dimethyl- [Y]	56	1010	*ms, lri*	104.76[ab]	77.37[a]	89.61[ab]	119.04[b]	4.298	0.004
3-Octene, 4-ethyl- [Y]	69	1012	*ms, lri*	29.19[ab]	20.38[a]	25.38[a]	38.69[b]	1.441	<0.001
Nonane, 3-methylene- [Y]	70	1022	*ms, lri*	236.74[ab]	188.34[a]	180.06[a]	284.91[b]	10.133	<0.001
Heptane, 2,2,4,6,6-pentamethyl- [Y]	57	1027	*ms, lri*	5140.73[a]	1929.11[a]	21626.35[b]	27733.83[c]	1183.950	<0.001
Decane [Y]	57	1030	*ms, lri, s*	406.72[c]	324.13[c]	225.36[b]	65.94[a]	16.694	<0.001
3-Ethyl-3-hexene [Y]	83	1042	*ms, lri*	62.24[ab]	47.97[a]	56.07[a]	78.99[b]	2.493	<0.001
1-Heptanol [YΦ]	70	1046	*ms, lri*	91.50[c]	36.91[a]	62.38[b]	71.79[bc]	3.218	<0.001
1-Octen-3-ol [YΦ]	57	1051	*ms, lri*	3935.68[c]	1915.73[a]	2824.65[b]	3607.07[c]	117.522	<0.001
5-Hepten-2-one, 6-methyl- [Y]	69	1056	*ms, lri*	128.81[b]	120.63[ab]	93.86[a]	116.58[ab]	3.997	0.011
2-Octanone [Y]	58	1059	*ms, lri*	41.33[ab]	38.50[a]	51.94[b]	72.99[c]	2.061	<0.001
Octanal [YΦ]	56	1066	*ms, lri, s*	384.48[b]	182.91[a]	209.96[a]	231.97[a]	12.175	<0.001
Undecane, 3,6-dimethyl- [Y]	57	1068	*ms, lri*	162.81[a]	83.59[a]	608.60[b]	879.39[c]	39.201	<0.001
Pentanoic acid [YΦ]	60	1083	*ms, lri*	394.31[b]	212.80[a]	257.50[a]	210.40[a]	13.552	<0.001
Undecane, 2,5-dimethyl- [Y]	57	1085	*ms, lri*	163.08[a]	142.14[a]	186.12[a]	258.59[b]	9.209	<0.001
Decane, 2,3,5-trimethyl- [Y]	57	1099	*ms, lri*	80.58[b]	76.68[b]	44.70[a]	70.74[b]	2.882	<0.001
Undecane [Y]	57	1113	*ms, lri, s*	1117.99[a]	1034.94[a]	1442.34[ab]	1864.94[b]	73.182	<0.001
2-Octenal, (E)- [YΦ]	70	1123	*ms, lri*	52.79[c]	9.63[a]	19.13[ab]	22.76[b]	1.993	<0.001
2,3-Dimethyl-3-heptene, (Z)- [Y]	83	1123	*ms, lri*	56.77[c]	15.27[a]	23.21[ab]	27.74[b]	1.972	<0.001

Compound									
1-Octanol [Υ][Φ]	56	1127	*ms, lri*	63.72[b]	43.46[a]	48.67[a]	48.86[a]	1.806	<0.001
Decanal [Υ][Φ]	81	1129	*ms, lri, s*	24.44[c]	14.33[a]	18.50[ab]	21.73[bc]	0.769	<0.001
2-Undecene, 9-methyl-, (Z)- [Υ]	70	1132	*ms, lri*	384.25[b]	344.35[ab]	275.11[a]	383.03[b]	13.208	0.007
3-Nonanone [Φ]	113	1134	*ms, lri*	21.19[a]	25.18[ab]	21.20[a]	29.87[b]	0.914	0.001
2-Nonanone [Φ]	58	1141	*ms, lri*	15.69[a]	24.12[b]	32.95[c]	46.71e	1.362	<0.001
5-Undecene, 6-methyl- [Υ]	168	1144	*ms, lri*	11.40[bc]	9.13[ab]	7.32[a]	12.51[c]	0.477	<0.001
Nonanal [Υ][Φ]	57	1148	*ms, lri, s*	538.49[b]	307.02[a]	303.28[a]	327.20[a]	14.872	<0.001
4,4-Dipropylheptane [Υ]	85	1153	*ms, lri*	55.24[b]	44.54[ab]	34.85[a]	43.85[ab]	1.804	0.001
5-Hexen-3-one [Φ]	57	1161	*ms, lri*	43.40[b]	35.58[ab]	27.78[a]	35.50[ab]	1.538	0.003
2-Undecene, 3-methyl-, (E)- [Υ]	70	1181	*ms, lri*	59.72	53.90	48.65	60.81	1.998	0.102
Dodecane [Υ]	57	1188	*ms, lri, s*	701.16[a]	663.91[a]	932.47[b]	1179.62[b]	41.201	<0.001
4-Nonene, 5-butyl- [Υ]	70	1197	*ms, lri*	24.54[b]	22.39[ab]	18.01[a]	20.65[ab]	0.857	0.043
4-Nonenal, (E)- [Υ]	83	1201	*ms, lri*	31.24[b]	18.32[a]	18.06[a]	23.63[a]	1.001	<0.001
Octanoic acid [Υ][Φ]	60	1224	*ms, lri*	30.74[c]	9.66[a]	21.75[b]	18.36[b]	1.197	<0.001
1-Tetradecanol [Υ][Φ]	68	1225	*ms, lri*	32.34[ab]	29.38[ab]	26.20[a]	34.48[b]	1.132	0.047
Decane, 3-ethyl-3-methyl- [Υ]	57	1228	*ms, lri*	50.73[b]	38.80[a]	32.66[a]	41.49[ab]	1.431	<0.001
1-Tetradecene [Υ]	97	1236	*ms, lri*	30.34[c]	23.99[ab]	19.00[a]	25.23[bc]	0.845	<0.001
Tridecane [Υ]	71	1258	*ms, lri, s*	190.43[b]	156.70[a]	245.19[c]	316.86[c]	11.383	<0.001
2-Decenal, (E)- [Φ]	70	1272	*ms, lri*	24.68[b]	12.77[a]	15.38[a]	16.81[a]	0.849	<0.001
2,4-Decadienal, (E,E)- [Υ][Φ]	81	1315	*ms, lri*	27.22[b]	4.07[a]	6.90[a]	7.73[a]	1.213	<0.001
2-Undecenal [Υ][Φ]	95	1339	*ms, lri*	5.96[b]	1.11[a]	1.42[a]	1.92[a]	0.266	<0.001
Pentadecanal- [Φ]	82	1516	*ms, lri, s*	2.15[a]	8.56[bc]	6.84[b]	10.40[c]	0.443	<0.001
Total lipolysis origin				45879.67[b]	19311.22[a]	47734.58[b]	64466.40[c]	1832.629	<0.001

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Compound	m/z	LRI	ID					SEM	p-value
Carbon disulfide [Y]	76	533	*ms, lri*	225.07[b]	119.20[a]	119.13[a]	148.43[a]	8.542	<0.001
Propanal, 2-methyl- [YΦ]	72	557	*ms, lri*	161.98[a]	225.54[b]	190.25[b]	241.26[b]	9.171	0.008
Fumaronitrile [Y]	78	646	*ms, lri*	29.18[c]	11.61[a]	10.27[a]	21.33[b]	0.948	<0.001
Butanal, 3-methyl- [YΦ]	58	659	*ms, lri*	1525.03[a]	1973.95[a]	1925.24[a]	2925.94[b]	86.149	<0.001
Butanal, 2-methyl- [YΦ]	57	671	*ms, lri*	758.92[a]	1291.93[b]	1195.03[b]	1278.51[b]	52.705	<0.001
2-Butenal, 2-methyl- [YΦ]	84	801	*ms, lri*	76.70[a]	63.38[a]	53.95[a]	106.33[b]	3.759	<0.001
1-Butanol, 3-methyl- [YΦΨ]	55	808	*ms, lri*	65.27[a]	425.21[c]	204.01[b]	240.17[b]	16.858	<0.001
1-Butanol, 2-methyl- [YΦ]	57	812	*ms, lri*	14.93[a]	51.65[c]	37.05[b]	44.65[bc]	1.890	<0.001
Propanoic acid, 2-methyl- [YΦ]	73	888	*ms, lri*	51.23[c]	18.99[a]	34.63[b]	60.85[c]	2.289	<0.001
2-Propanol, 2-methyl- [YΦ]	59	894	*ms, lri*	17.68[c]	6.80[a]	7.49[a]	12.68[b]	0.556	<0.001
3-(1'-pyrrolidinyl)-2-butanone [Y]	98	906	*ms, lri*	136.09[b]	83.70[a]	74.19[a]	94.24[a]	4.904	<0.001
3-Pentanol, 2,4-dimethyl- [YΦ]	73	954	*ms, lri*	7.84[a]	8.75[ab]	7.43[a]	10.29[b]	0.236	<0.001
Butanoic acid, 3-methyl- [YΦ]	60	969	*ms, lri*	349.62[c]	139.30[a]	250.02[b]	327.58[bc]	14.309	<0.001
Pyrazine, 2,6-dimethyl- [YΦ]	108	978	*ms, lri*	290.52[b]	344.23[c]	241.18[a]	395.39[c]	10.530	<0.001
1-(1'-pyrrolidinyl)-2-butanone [Y]	84	982	*ms, lri*	129.87[bc]	84.97[a]	98.05[ab]	138.36[c]	5.375	0.001
Dimethyl trisulfide [YΦ]	126	1035	*ms, lri*	179.23[b]	13.88[a]	7.19[a]	11.39[a]	8.662	<0.001
Benzaldehyde [YΦ]	106	1045	*ms, lri*	339.48[a]	350.30[a]	295.39[a]	462.27[b]	11.745	<0.001
1-Heptanol, 2,4-diethyl- [YΦ]	69	1085	*ms, lri*	90.78[ab]	73.41[a]	69.19[a]	108.27[b]	3.947	0.001
2-Ethyl-1-hexanol [YΦ]	57	1094	*ms, lri*	6.65[a]	58.12[b]	128.81[d]	86.52[c]	5.083	<0.001
5-Ethylcyclopent-1-enecarboxaldehyde	124	1099	*ms, lri*	27.85[b]	10.17[a]	13.43[a]	14.93[a]	0.934	<0.001
2(3H)-Furanone, 5-ethyldihydro- [YΦ]	85	1158	*ms, lri*	174.77[a]	197.82[b]	178.62[b]	215.24[b]	5.130	0.014
4-Methyl-5-decanol [YΦ]	55	1162	*ms, lri*	21.99[b]	12.83[a]	15.13[a]	17.55[ab]	0.750	<0.001
Sulfurous acid, butyl dodecyl ester [YΦ]	85	1304	*ms, lri*	27.37[b]	28.21[b]	18.48[a]	27.32[b]	0.772	<0.001

				4708.05[a]	5593.97[a]	5174.15[a]	6989.50[b]	154.115	<0.001
Total proteolysis origin									
Pentane, 2-methyl-[Y][Φ]	71	543	*ms, lri*	2.61[a]	1.20[a]	2.75[a]	13.89[b]	0.559	<0.001
Acetic acid ethenyl ester [Y]	86	588	*ms, lri*	25.79[bc]	21.53[b]	14.90[a]	28.64[c]	0.898	<0.001
Ethyl Acetate [Y]	61	598	*ms, lri*	148.15[a]	238.69[b]	213.97[ab]	215.97[ab]	10.382	0.013
Methane, oxybis[dichloro-[Y]	83	611	*ms, lri*	270.11[b]	121.20[a]	318.55[b]	455.68[c]	16.324	<0.001
Propanoic acid, ethyl ester [Y]	57	737	*ms, lri*	52.51[c]	18.56[a]	30.57[ab]	42.12[bc]	2.190	<0.001
Disulfide, dimethyl [Φ]	94	781	*ms, lri*	1786.20[b]	160.12[a]	61.83[a]	213.32[a]	77.445	<0.001
Butanoic acid, ethyl ester [Y]	71	855	*ms, lri*	86.10[a]	78.86[a]	68.63[a]	136.66[b]	4.090	<0.001
Octane, 2-methyl-[Y][Φ]	71	899	*ms, lri*	16.79[c]	10.29[a]	11.93[ab]	15.87[bc]	0.627	<0.001
Butanoic acid, 2-methyl-, ethyl ester [Y]	102	908	*ms, lri*	49.05[a]	65.69[ab]	56.95[a]	85.20[b]	2.925	<0.001
Butanoic acid, 3-methyl-, ethyl ester [Y]	88	913	*ms, lri*	130.87[a]	170.48[a]	153.26[a]	280.27[b]	11.318	<0.001
Oxalic acid, butyl propyl ester [Y]	57	936	*ms, lri*	201.88[c]	123.93[b]	57.32[a]	84.50[ab]	7.840	<0.001
Ethanol, 2-butoxy-[Y]	57	985	*ms, lri*	431.27[a]	353.25[a]	797.03[b]	947.37[c]	29.190	<0.001
Carbonic acid, bis(2-ethylhexyl) ester [Y]	112	1003	*ms, lri*	30.15[b]	24.44[ab]	17.32[a]	23.12[ab]	1.093	<0.001
Hexanoic acid, ethyl ester [Y]	88	1050	*ms, lri*	167.48[a]	205.07[a]	205.07[a]	274.88[b]	7.699	<0.001
Tridecane, 6-methyl-[Y][Φ]	57	1079	*ms, lri*	323.72[a]	207.70[a]	636.89[b]	884.36[c]	35.976	<0.001
2-Piperidinecarboxylic acid, 1-acetyl-, ethyl ester [Y]	84	1124	*ms, lri*	36.93[c]	12.69[a]	15.86[ab]	20.18[b]	1.073	<0.001
Octanoic acid, ethyl ester [Y]	88	1204	*ms, lri*	74.38	80.58	73.08	68.96	1.749	0.149
Dodecane, 2-methyl-[Y][Φ]	88	1233	*ms, lri*	22.03[a]	23.92[a]	40.66[b]	53.41[c]	2.015	<0.001
Tridecane, 3-methyl-[Y][Φ]	85	1304	*ms, lri*	27.62[b]	28.46[b]	18.60[a]	27.30[b]	0.823	<0.001
Decanoic acid, ethyl ester [Y]	88	1336	*ms, lri*	28.59[c]	20.50[b]	12.30[a]	16.53[ab]	0.860	<0.001
2,2,4-Trimethyl-1,3-pentanediol diisobutyrate [Y]	71	1442	*ms, lri*	10.00[a]	5.82[a]	2.64[a]	58.11[b]	2.772	<0.001
Total microbial origin				3922.23[c]	1972.97[a]	2810.12[b]	3946.33[c]	97.439	<0.001

Compound	m/z	LRI	R	CO	HPP-0	HPP-20	HPP-35	SEM	P
Acetoin	45	787	*ms, lri*	478.36[b]	299.84[a]	367.90[ab]	625.50[c]	19.901	<0.001
Cyclobutane, 1,1,2,3,3-pentamethyl-	70	813	*ms, lri*	326.38[b]	172.20[a]	402.58[b]	569.07[c]	20.318	<0.001
Ethylbenzene	91	917	*ms, lri*	21.49[bc]	18.32[b]	13.48[a]	21.96[c]	0.538	<0.001
Benzene, 1,3-dimethyl-	106	926	*ms, lri*	27.03[c]	24.88[bc]	18.01[a]	22.00[ab]	0.647	<0.001
Cyclohexanone, 2-ethyl-	69	972	*ms, lri*	62.10[ab]	44.11[a]	44.04[a]	70.01[b]	3.140	0.004
4-Octanone, 5-hydroxy-2,7-dimethyl-	69	1042	*ms, lri*	13.29[bc]	9.05[a]	10.38[ab]	14.64[c]	0.447	<0.001
4-Ethylcyclohexanol	81	1104	*ms, lri*	120.90[b]	86.88[a]	106.36[ab]	118.21[b]	3.718	0.006
Benzeneacetaldehyde	91	1119	*ms, lri*	712.67[b]	514.37[a]	501.69[a]	680.17[b]	18.764	<0.001
Cyclopentane, 1-ethyl-3-methyl-	83	1123	*ms, lri*	56.77[c]	15.27[a]	23.21[ab]	27.74[b]	1.972	<0.001
Benzyl alcohol	108	1124	*ms, lri*	125.21[a]	291.08[b]	425.52[c]	552.39[d]	17.344	<0.001
1-Hexanone, 5-methyl-1-phenyl-	105	1137	*ms, lri*	11.53[a]	11.66[a]	34.01[b]	75.99[c]	2.754	<0.001
Cyclopentane, ethyl-	98	1148	*ms, lri*	261.83[b]	134.68[a]	142.63[a]	156.46[a]	7.681	<0.001
p-Cresol	107	1178	*ms, lri*	29.90[ab]	23.63[a]	27.27[a]	33.88[b]	0.908	0.001
Phenylethyl Alcohol	92	1182	*ms, lri*	10.55[a]	45.80[b]	14.56[a]	17.64[a]	1.766	<0.001
Benzaldehyde, 3-ethyl-	134	1209	*ms, lri*	34.40[c]	14.22[a]	21.36[b]	27.37[b]	1.077	<0.001
Total unknoun origin				**2292.41[b]**	**1705.98[a]**	**2153.01[b]**	**3013.04[c]**	**56.635**	**<0.001**
Total compounds				**56802.37[b]**	**28584.14[a]**	**57871.86[b]**	**78415.27[c]**	**1973.358**	**<0.001**

Compound origin according to: [ϒ] Narváez-Rivas et al. (2012) [Φ] Martín et al. (2006) [Ψ] Fonseca et al. (2015). [a-d] Mean values in the same row (corresponding to the same compound) not followed by a common letter differ significantly ($P<0.05$; Tukey´s Test). SEM: standard error of mean.

m/z: Quantification ion; LRI: Lineal Retention Index calculated for DB-624 capillary column (J&W scientific: 30m×0.25mm id, 1.4μm film thickness) installed on a gas chromatograph equipped with a mass selective detector; R: Reliability of identification; *lri*: linear retention index in agreement with literature (Domínguez et al., 2014; Lorenzo, Montes, Purriños, & Franco, 2012; Lorenzo, Bedia, & Bañon, 2013; Lorenzo, 2014; Lorenzo & Domínguez, 2014; Lorenzo & Carballo, 2015; Pateiro, Franco, Carril, & Lorenzo, 2015; Pérez-Santaescolástica et al., 2018a; Pérez-Santaescolástica et al., 2018b; Purriños, Franco, Bermudez, Carballo, & Lorenzo, 2011a; Purriños, Franco, Bermúdez, Temperan, Carballo, & Lorenzo, 2011b; Purriños, Franco, Carballo, & Lorenzo, 2012, Purriños, Carballo, & Lorenzo, 2013); *ms*: mass spectrum agreed with mass database (NIST14); *s*: mass spectrum and retention time identical with an authentic standard.

Treatments: CO= control (without treatment); HPP-0=High pressure treatment at 0 ºC; HPP-20=High pressure treatment at 20 ºC; HPP-35=High pressure treatment at 35 ºC.