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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<0.001) 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<0.001) 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 ºC, except for isoleucine which presented the highest level in samples from CV group. Similarly, significant differences (P<0.05) were also detected in the total volatile compound content between CO and US groups, with a higher concentration in the CO batch (56662.84 AU x 10^3 / g of dry-cured ham) than in the US treatment (45848.47 AU x 10^3 / g of dry-cured ham), being the values in the CV treatment intermediate (48497.25 AU x 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.

Keywords: adhesiveness; dry-cured ham; free amino acid content; heat treatment; proteolysis; ultrasound treatment; volatile 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, Gómez, & Domínguez, 2015a). 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 (Skrlep *et al.,* 2011). López-Pedrouso *et al.* (2018) noticed that the determination of instrumental adhesiveness could be a good indicator 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 ºC) during a long time (between 7 and 10 days) have been used to correct the softness and pastiness of dry-cured ham (Morales, Arnau, Serra, Guerrero, & Gou, 2008; Gou, Morales, Serra, Guardia, & Arnau, 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 García-Pérez (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 ºC 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 ºC) 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 hours at 50 ºC.

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 Lopez-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

155 to a series of standard alkanes (C_5–C_{14}) (for calculating Kovats indexes, Supelco 44585-U,
156 Bellefonte, PA, USA) and matching them with data reported in literature. The results are
157 expressed as quantified area units (AU) × 10^3/g of sample.

158 *2.6. Statistical analysis*

159 The effect of treatment was examined using a one-way ANOVA, where this parameter was
160 set as factor. The values were given in terms of mean values and standard error of the means
161 (SEM). When a significant effect (P<0.05) was detected, means were compared using the Tukey´s
162 test. All analyses were conducted using the IBM SPSS Statistics 24.0 program (IBM Corporation,
163 Somers, NY, USA) software package. Correlations between variables (P<0.05) were determined
164 using the Pearson's linear correlation coefficient.

165 **3. Results and discussion**

166 *3.1. Effect of treatments on instrumental adhesiveness*

167 The effect of temperature treatment alone or US assisted on instrumental adhesiveness of
168 dry-cured ham is shown in Figure 1. Statistical analysis showed that both, US and CV treatments,
169 significantly (P<0.001) decreased the instrumental adhesiveness of dry-cured hand from 85.27
170 g for CO to 40.59 and 38.68 g for US and CV groups, respectively. However, there was not
171 significant differences between US and CV treatments. The decrease of instrumental
172 adhesiveness in dry-cured ham slices may be due to the fact that the intramolecular hydrogen
173 connections can break due to the mechanical vibration and the effects of thermal and ultrasonic
174 cavitation causing loosening of the molecular structure and reduction of molecular nodes (Luo,
175 Huang, Yang, 2003). In addition, denaturation and structural changes of proteins due to thermal
176 treatment could also decrease the instrumental adhesiveness of dry-cured ham slices (Tornberg,
177 2005). Finally, some changes such as the aggregation of the globular heads of myosin (Morales
178 *et al.,* 2008), cell membrane destruction (Rowe, 1989) and the transversal and longitudinal
179 shrinkage of meat fibers (Tornberg, 2005) could take place during the thermal treatment.

180 The findings in the present work are in agreement with data reported by Morales *et al.*
181 (2008) who showed that the thermal treatment at 30 ºC for 168 h on both sliced and whole dry-

cured ham decreased softness, adhesiveness and pastiness in BF muscle, without increasing hardness in SM muscle or affecting their physicochemical parameters (moisture, activity water and proteolysis index). In addition, Gou *et al.* (2008) observed a decrease of soft textures in whole dry-cured ham pieces without affecting the sensory properties after a treatment of 10 days ageing process at 30 ºC. Regarding US application, our outcomes are in agreement with data reported by Contreras *et al.* (2018) who did not find any significant difference in hardness and elasticity of dry-cured ham slices between ultrasonically assisted heated and conventionally heated samples. However, our results are in disagreement with those reported by Hu *et al.* (2014) who did not show significant difference between control and US starch corn samples, but they found a lower hardness, elasticity and brittleness in US treated samples.

Taking into account that texture is one the most important sensory attributes of dry-cured ham, which affect its acceptability by consumer, the application of both treatments, US and CV, could be used to reduce the instrumental adhesiveness of dry-cured ham slices by immersing the packaged samples in a water bath during a short period of time.

3.2. Effect of treatments on moisture content

The effect of temperature treatment alone or US assisted on moisture content is presented in Figure 2. Statistical analysis did not show significant differences on moisture content among groups, presenting mean values of 59.01, 58.68 and 58.57 g/100 g; $P>0.05$, for CO, US and CV groups, respectively. Our moisture values were in the range of data (48.3-65.2 g/100 g) reported by other authors (Bermúdez, Franco, Carballo, & Lorenzo, 2014a; Prevolnik *et al.,* 2011; Pugliese *et al.,* 2015) for dry-cured ham.

3.3. Effect of treatments on free amino acid content

Table 1 shows the effect of temperature treatment alone or US assisted on the free amino acids of dry-cured ham. Statistical analysis displayed that total free amino acid content was significantly ($P<0.001$) affected by both treatments, presenting the 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. These

values are within the range of free amino acid contents (from 4000 to 12,500 mg/100 g dry matter) described by other authors (Bermúdez, Franco, Carballo, Sentandreu, & Lorenzo, 2014b; Jurado, García, Timón, & Carrapiso, 2007; Martín, Antequera, Ventanas, Benítez-Donoso, & Córdoba, 2001) in dry-cured ham. The higher total free amino acid content in samples submitted to ultrasound at 50 ºC could be due to the release of some free amino acids from cell tissues that were destroyed by the ultrasounds.

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 ºC, except for isoleucine which presented the highest level in samples from CV group. According to Jambrak, Mason, Lelas, Paniwnyk, & Herceg (2014), the ultrasound treatment can modify the protein structure due to partial cleavage of intermolecular hydrophobic interactions, rather than peptide or disulphide bonds increased the release of free amino acids. It could be seen that leucine, glutamic acid and alanine were the most abundant free amino acid in the three studied groups and the sum of these three amino acids reached around 27% of the total free amino acids.

On the other hand, the flavour of dry-cured ham could be linked to the amount of the individual free amino acid. In this regard, sweet taste is associated with the level of alanine, serine, proline, threonine and glycine; bitter taste is related to aromatic amino acids such as leucine, phenylalanine, methionine, valine and isoleucine; whereas acid taste is linked to histidine, glutamic and aspartic acids, and aged flavour is associated with the content of lysine, tyrosine and aspartic acid (Table 1). According to this classification, both treatments (ultrasound and temperature) significantly increased the bitter taste of dry-cured ham. On the other hand, the use of temperature did not significantly modify the acid and aged taste, whereas these two tastes were significantly increased by using ultrasounds. The temperature significantly increased the sweet taste of hams and this taste was significantly further increased by the ultrasound treatment at 50 ºC. These variations in free amino acid content could be affected the acceptance of dry-cured ham for the consumers.

3.4. Effect of treatments on volatile compound profile

The effect of temperature treatment alone or US assisted on the volatile fraction of dry-cured ham can be observed in Table 2. A total of 155 volatile compounds were found in headspace of the dry-cured ham. These volatile compounds were classified as part of some of the main chemical families according to Narváez-Rivas *et al.* (2012) and Purriños, Franco, Bermúdez, Carballo and Lorenzo (2011a): 56 hydrocarbons, 23 aldehydes, 21 ketones, 16 esters and ethers, 24 alcohols, 6 carboxylic acids, 4 nitrogenous compounds and 5 sulphur compounds. Significant differences (P<0.05) were detected in the total volatile compound content between CO and US groups, with a higher concentration in the CO batch (56662.84 AU x 10^3 / g of dry-cured ham) than in the US treatment (45848.47 AU x 10^3 / g of dry-cured ham), being the values in the CV treatment intermediate (48497.25 AU x 10^3 / g of dry-cured ham). The fact that US had been used as a method to improve the food preservation (Knorr *et al.,* 2011) together with the hypothesis that spoilage could originate higher concentrations of volatile compounds in the headspace (Carrapiso, Martín, Jurado, & García, 2010), could explain the less content of total volatile compounds in the US group. Regarding the different chemical families, except for hydrocarbons, the sum of the volatile compounds of each family showed significant differences among groups. Moreover, the levels of 94 individually volatile compounds were significantly influenced by the treatment (24 hydrocarbons, 15 ketones, 15 alcohols, 21 aldehydes, 10 ester and ethers, 4carboxilic acids, 3 sulfur compounds and 2 nitrogenous compounds).

As shown in Table 2, hydrocarbons were the most numerous chemical family with up to 56 different compounds, 24 of them have already been identified in other previous studies in hams (Bermúdez, Franco, Carballo, & Lorenzo, 2015; Narváez-Rivas *et al.,* 2012; Pérez-Santaescolástica *et al.,* 2018). Hydrocarbons represented a percentage of 30% of the total area of the volatile compounds in control samples, whereas, in both US and CV groups, this chemical family was the most abundant (accounting for 43% and 37%, for US and CV batches, respectively). The aliphatic hydrocarbon, that was found in higher concentration was 2,2,4,6,6-pentamethyl heptane, followed by octane, and then, with similar values, pentane, hexane,

undecane and dodecane. It is well known that significant differences in the hydrocarbons content does not originate important odour changes due to their low threshold values (Carrapiso, Ventanas, & García, 2002).

Meanwhile, the main family of volatile compounds in CO group were the aldehydes (approximately 41% of the total area of volatile compounds). In this regard, Garcia *et al.* (1991) identified linear aldehydes as a secondary product of lipid oxidative decomposition and attributed the origin of branched aldehydes to non-enzymatic Strecker degradation of valine, leucine and isoleucine. In our work an important reduction of total aldehydes content in US group was observed, as well as a higher decrease in CV batch (23509.08 *vs*. 10307.72 *vs*. 2381.68 AU x 10^3 / g of dry-cured ham for CO, US and CV groups, respectively). According with previous studies in ham (Andres, Cava, Ventanas, Muriel, & Ruiz, 2007; García-González, Tena, Aparicio-Ruiz, & Morales, 2008; Garcia *et al.,* 1991; Jurado, Carrapiso, Ventanasa, & García, 2009; Sánchez-Peña, Luna, García-González, & Aparicio, 2005), hexanal was the predominant linear aldehyde in CO and US groups, with the highest content presented in CO samples (12264.83 *vs*. 5747.78 *vs*. 185.78 AU x 10^3 / g of dry-cured ham for CO, US and CV groups, respectively). Hexanal is considered the main volatile compound derived from oxidation of n-6 fatty acids such as linoleic and arachidonic acids, which contributes to the green, greasy and fatty distinctive flavour in matured hams (García González, Tena, Aparicio-Ruiz, & Morales, 2008). In contrast, CV batch presented propanal as the main aldehyde, whose concentration was higher than in the other two groups. On the other hand, 3-methyl butanal was the most abundant branched aldehyde determined in all cases but presenting significant differences (P<0.001) among the groups. CO samples showed the highest concentration of this compound, while CV group registered the lowest one. In this way, Pérez-Santaescolástica *et al.* (2018) found that high-proteolytic hams presented lower amounts of hexanal and 3-methyl butanal than low-proteolytic hams. Lower amounts of these aldehydes in both treatment groups than in control was expected since high temperatures promote protein degradation and enhance proteolytic reactions. According to Ramirez & Cava (2007), who proposed the degradation of isoleucine amino acid as

290 the most probably origin of 2-methyl butanal, a negative correlation between these compounds
291 was found (r= -0.547; *P*<0.01), as well as significant (*P*<0.001) difference among the groups,
292 obtaining higher levels in CV group than in the others ones.

293 Likewise, the total alcohol content showed higher levels in CV samples than in the other
294 two groups (6548.61 *vs*. 8599.43 *vs*. 12199.24 AU x 10^3 / g of dry-cured ham for CO, US and CV
295 groups, respectively). This high content of total alcohols found in CV group is a consequence of
296 the higher amounts of three specific individual alcohols: 2-methyl butanol, 3-methyl butanol and
297 phenylethyl alcohol. The increment of 2-methyl butanol and 3-methyl butanol in CV group could
298 be explained for the decrease observed in the 2-methyl butanal and 3-methyl butanal since that
299 branches alcohols may be originated, among others reasons, from the reduction of branched
300 aldehydes (Martín, Córdoba, Aranda, Córdoba, & Asensio, 2006). Otherwise, the major alcohol
301 detected in similar levels in all the groups was 1-octen-3-ol (3543.17 *vs*. 3818 *vs*. 3922.68 AU x
302 10^3 / g of dry-cured ham for CO, US and CV groups, respectively).

303 In addition to aldehydes, Carrapiso, Ventanas, & García (2002) identified ketones as
304 important compounds to odour contribute in dry-cured ham. In our study, statistical analysis
305 showed that the total ketones content was significantly (*P*<0.001) affected by the treatment,
306 observing the greatest level in CV group, and being the 2-heptanone and the acetoin the most
307 abundant ones with higher amount in CV samples than in CO and US groups (427.95 *vs*. 664.14
308 *vs*. 980.43 and 484.130 *vs*. 501.60 *vs*. 231.51 AU x 10^3 / g of dry-cured ham for CO, US and CV
309 groups, respectively). In agreement with previous studies (Ramírez & Cava, 2007; Sabio, Vidal-
310 Aragón, Bernalte, & Gata, 1998), other 2-ketones were also found, such as 2-butanone, 2-
311 pentanone, 2-octanone and 2-nonanone. All these compounds presented the highest values in
312 the samples from CV treatment.

313 Esters and ethers, carboxylic acids, nitrogenous compounds and sulfur compounds were
314 the chemical families that presented minor levels of volatile compounds. Esters are compounds
315 distributed in the essential oils with a high flavouring effects, derived from the reaction of an
316 alcohol or phenol with acids (Reineccius, 1991). Some studies reported low values of esters in

317 volatile dry-cured ham profiles (Martín *et al.,* 2006), whereas other studies carried out in cooked
318 pork meat showed a greater content of these compounds (Gorbatov & Lyaskovskaya, 1980).
319 According to this, it could be assumed that temperature affects the ester compound formation.
320 However, this effect was not observed in the present study, since the CV samples showed the
321 lowest total content of esters (1906.99 *vs*. 1680.82 *vs*.1385.33 AU x 10^3 / g of dry-cured ham for
322 CO, US and CV groups, respectively). This fact may be explained because the high temperature
323 produced losses by volatilisation.

324 Regarding carboxylic acids, total content was 20% less in US group and 70% in CV
325 treatment than in CO group. The highest differences were found between pentanoic acid and
326 butanoic acid contents.

327 On the other hand, 2,6-dimethyl pyrazine was found as the main nitrogenous compound.
328 Pyrazines are usual compounds in meat and meat products cooked at high temperatures
329 (Mussinan & Walradt, 1974), and their formation is a result of the reaction between diketones
330 and amino compounds at high temperatures (Shibamoto & Bernhard, 1976). According to this,
331 CV samples showed higher significant values (P<0.001) than the other batches, whereas US
332 batch did not show any difference compared with CO group. It is possible that the structural
333 changes that were originated by US application can prevent reactions between diketones and
334 amino compounds.

335 Finally, the temperature application also originated an important decrease in the sulfur
336 compounds, being the dimethyl disulfide the most affected compound (1740.04 *vs*. 206.48 *vs*.
337 738.87 AU x 103 / g of dry-cured ham for CO, US and CV groups, respectively). The sulfur amino
338 acids showed a negative and significant (P<0.01) correlation with dimethyl disulfide (r = -0.557,
339 r = -0.614 and r = -0.512, for taurine, cysteine and methionine, respectively) and dimethyl
340 trisulfide (r = -0.550, r = -0.599 and r = -0.493, for taurine, cysteine and methionine, respectively),
341 suggesting that these compounds could be originated by the amino acids catabolism (Sabio *et*
342 *al.,* 1998).

343 *3.5. Effect of treatment on* sensory attributes

344 It is worth noting that not all the volatile compounds contribute in the same way to the final
345 odour because only a small percentage of them are odour active and the sensory characteristics
346 can change depending on their concentrations and on the synergies with other compounds of
347 the matrix (Aparicio & Morales, 1998). Over the years, some authors have investigated the
348 relationship between volatile compounds and the odour characteristics (Carrapiso *et al.,* 2010;
349 García-González *et al.,* 2008; Narváez-Rivas *et al.,* 2012). In this context, Figure 3 shows the
350 most odour compounds in dry-cured ham identifying and comparing their contents in the different
351 treatments. Due to different amounts, selected sensory descriptors related to each volatile
352 compound were grouped in three intervals for a better comprehension: A (0-15000 AU x 10^3 / g
353 of dry-cured ham), B (0-2000 AU x 10^3 / g of dry-cured ham) and C (0-400 AU x 10^3 / g of dry-
354 cured ham).
355 In case of the hydrocarbons, only five compounds were previously described as odour
356 descriptors, octane, heptane, hexane, ethyl benzene and 2-ethyl furan, whose contribution is
357 related with sweet notes. As mentioned above, this chemical family has not very odorant impact,
358 because of its high threshold. Considering their low threshold, aldehydes are the most intensive
359 compounds followed by ketones and esters, and to a lesser extent by alcohols. Hexanal and 3-
360 methyl butanol are the most odour-active compounds identified in hams (Carrapiso *et al.,* 2002)
361 and were the main volatile compounds showed in CO samples, contributing principally with the
362 characteristic greasy odour of ham and to a lesser extent with fruity notes. Significant lower levels
363 of hexanal were found in treated groups, observing the lowest content in CV group. Lower
364 contents in CV batch also detected for nonanal, octanal, heptanal, 2-methyl butanal, 3-methyl
365 butanal, 2,4-decadienal, 4-nonenal, 2-octenal 2-methyl propanal, methional and benzaldehyde.
366 According to this, the application of high temperature without ultrasound could promote an
367 important reduction, specially, on fatty and grassy notes. Regarding ketones, the CV group
368 presented higher levels in four of the six odour active ketones found in this study, so the odour
369 of this group of hams could be more floral and fruity compared with the others. On the other
370 hand, alcohols with a low molecular weight confer a sweet and spirituous odour to ham, but as

371 the molecular weight increases a fatty and irritating odour is perceived (Narváez-Rivas *et al.,*
372 2016). Samples from CV group showed higher values of 3-methyl butanol, compound associated
373 to biceps femoris muscle (Sánchez-Peña *et al.,* 2005), and 2-butanol than the other two groups.
374 Additionally, it was observed fatty, balsamic and fruity notes reduction due to the lowest amounts
375 of pentanol, octanol and butanol presented in these samples. It was not found significant
376 differences in 1-octen-3-ol among the groups, a fact that was expected since this compound that
377 contributes with a typical mushroom odour is derived from feeding system (Jurado *et al.,* 2009).
378 Among the esters reported in previous studies, only one was detected here. Ethyl ester butanoic
379 acid was identified as a specific odour-active compound in Iberian (Carrapiso *et al.,* 2010),
380 Serrano (Flores, Grimm, Toldrá, & Spanier, 1997) and Jinhua (Song, Cadwallader, & Singh,
381 2008) hams.
382 Finally, dimethyl disulfide and some carboxylic acids (butanoic, propanoic, pentanoic and
383 3-methyl butanoic acid) were previously reported like spoiled ham odorants (Carrapiso, Martín,
384 Jurado, & García, 2010). In this context, CO group showed higher spoiled and rancid odour due
385 to its higher amounts of butanoic, pentanoic, 3-methyl butanoic acid and dimethyl disulfide (see
386 Figure 3b and 3c).
387 **4. Conclusions**
388 The thermal treatment (5 hours at 50 ºC) of sliced, vacuum packaged high proteolysis hams
389 applied both alone and assisted by ultrasonic treatment during the first 7.5 minutes of thermal
390 treatment significantly decreased the adhesiveness of hams. However, both treatments
391 significantly affected the total and individual free amino acid content. These treatments had also
392 a significant effect on the total volatile compounds and on the contents of the different families of
393 volatiles. Taking into account the specific taste of some free amino acids and also the particular
394 aroma notes of the different volatile compounds, and despite the limitations of the present work
395 (no quantification or normalization was done for the extraction of volatile molecules and sensorial
396 analyses were not carried out), an effect of these two treatments on the taste and odor of ham
397 could be expected.

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579

Caption to figures

Figure 1. Effect of temperature treatment alone (CV) or US assisted (US) on instrumental adhesiveness of dry-cured ham. Plotted values are means and standard deviations of the results from twenty-six samples of each group

Figure 2. Effect of temperature treatment alone (CV) or US assisted (US) on moisture content of dry-cured ham. Plotted values are means and standard deviations of the results from twenty-six samples of each group

Figure 3. Comparative sensory descriptors among treatments. Sensory descriptions are given in agreement with: Garcia Gonzalez *et al.* (2008), Carrapiso *et al.* (2010); Carrapiso *et al.* (2002) and Narváez-Rivas *et al.* (2012). Selected sensory descriptors related to each volatile compound were grouped in three intervals for a better comprehension: A (0-15000AU x 10^3 / g of dry-cured ham), B (0-2000AU x 10^3 / g of dry-cured ham) and C (0-400 AU x 10^3 / g of dry-cured ham.

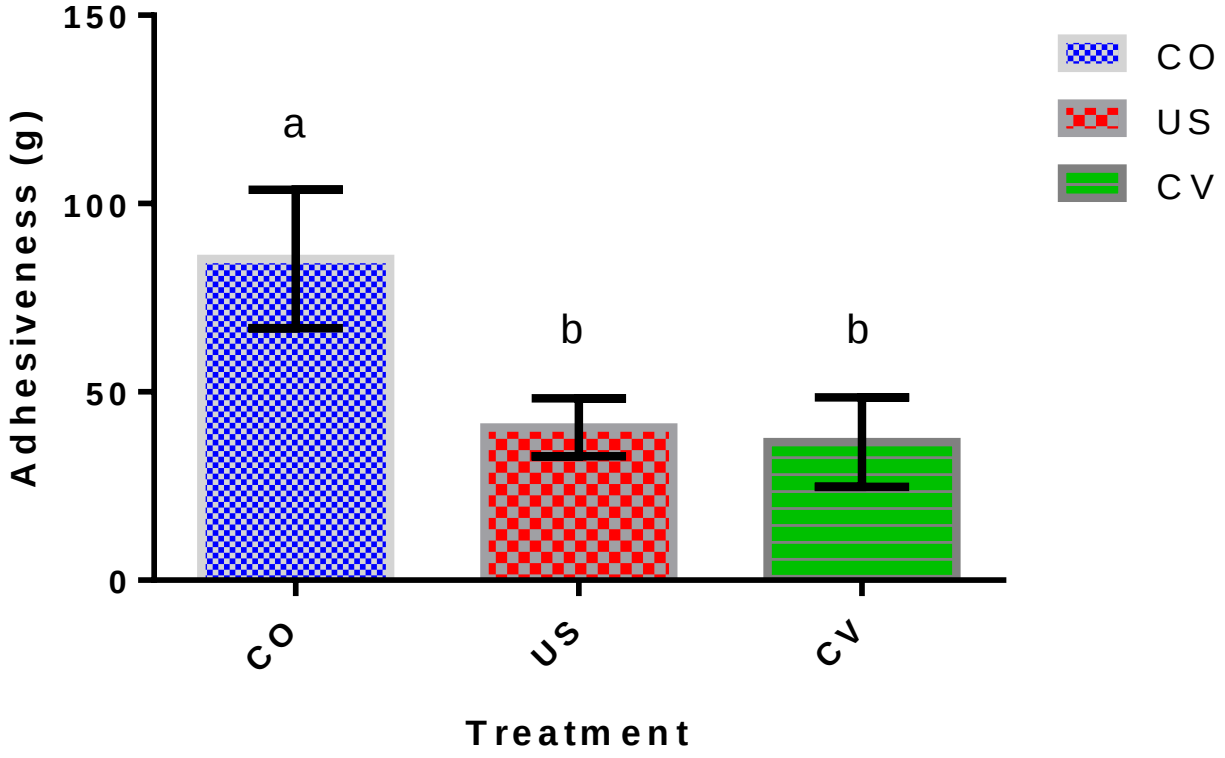


Figure 1

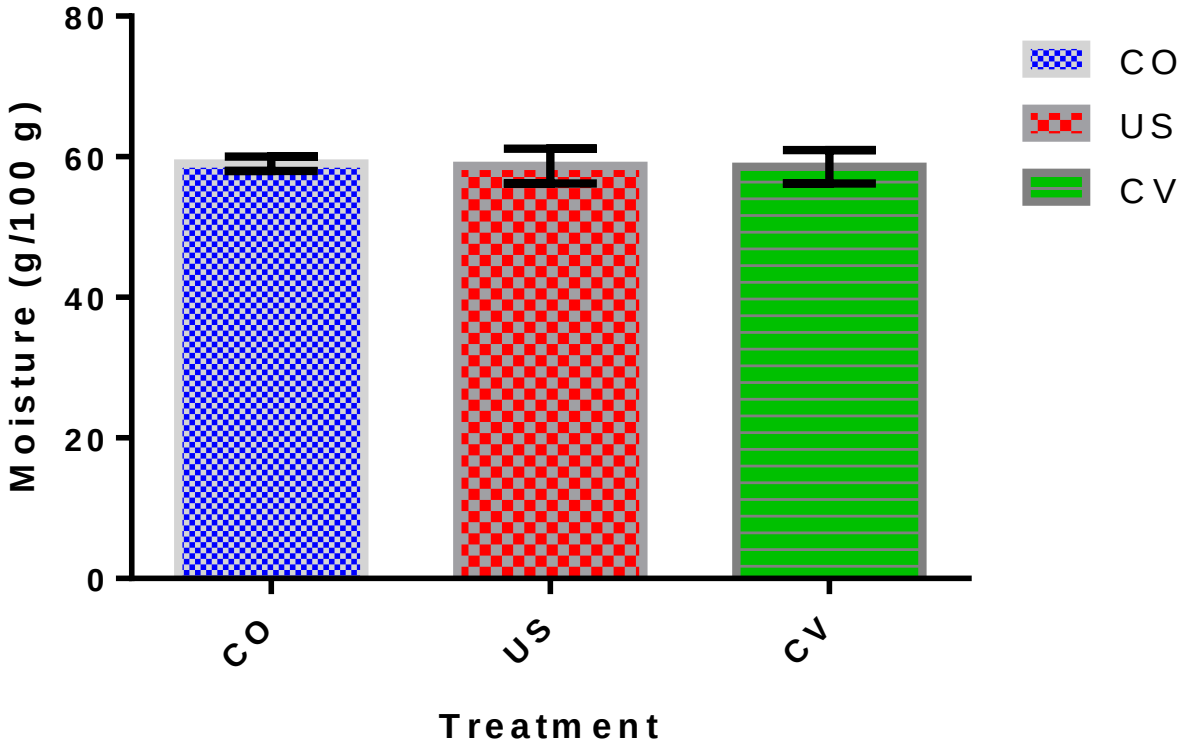
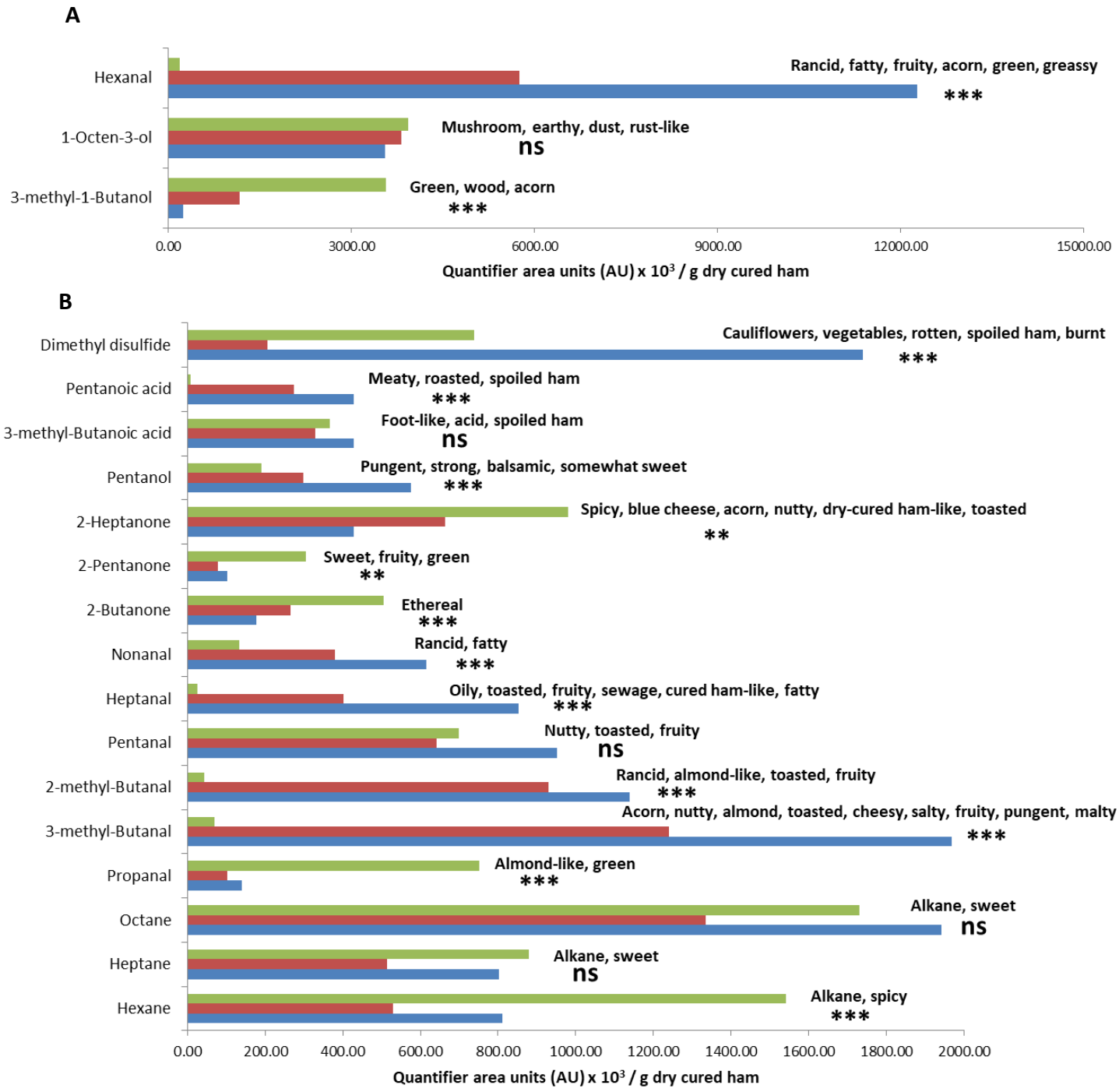


Figure 2


A
Hexanal
Rancid, fatty, fruity, acorn, green, greassy
***
1-Octen-3-ol
Mushroom, earthy, dust, rust-like
ns
3-methyl-1-Butanol
Green, wood, acorn
***
0.00
3000.00
6000.00
9000.00
12000.00
15000.00
Quantifier area units (AU) x 10³ / g dry cured ham
B
Dimethyl disulfide
Cauliflowers, vegetables, rotten, spoiled ham, burnt
***
Pentanoic acid
Meaty, roasted, spoiled ham
***
3-methyl-Butanoic acid
Foot-like, acid, spoiled ham
ns
Pentanol
Pungent, strong, balsamic, somewhat sweet
***
2-Heptanone
Spicy, blue cheese, acorn, nutty, dry-cured ham-like, toasted
**
2-Pentanone
Sweet, fruity, green
**
2-Butanone
Ethereal
***
Nonanal
Rancid, fatty
***
Heptanal
Oily, toasted, fruity, sewage, cured ham-like, fatty
***
Pentanal
Nutty, toasted, fruity
ns
2-methyl-Butanal
Rancid, almond-like, toasted, fruity
***
3-methyl-Butanal
Acorn, nutty, almond, toasted, cheesy, salty, fruity, pungent, malty
***
Propanal
Almond-like, green
***
Octane
Alkane, sweet
ns
Heptane
Alkane, sweet
ns
Hexane
Alkane, spicy
***
0.00
200.00
400.00
600.00
800.00
1000.00
1200.00
1400.00
1600.00
1800.00
2000.00
Quantifier area units (AU) x 10³ / g dry cured ham

C

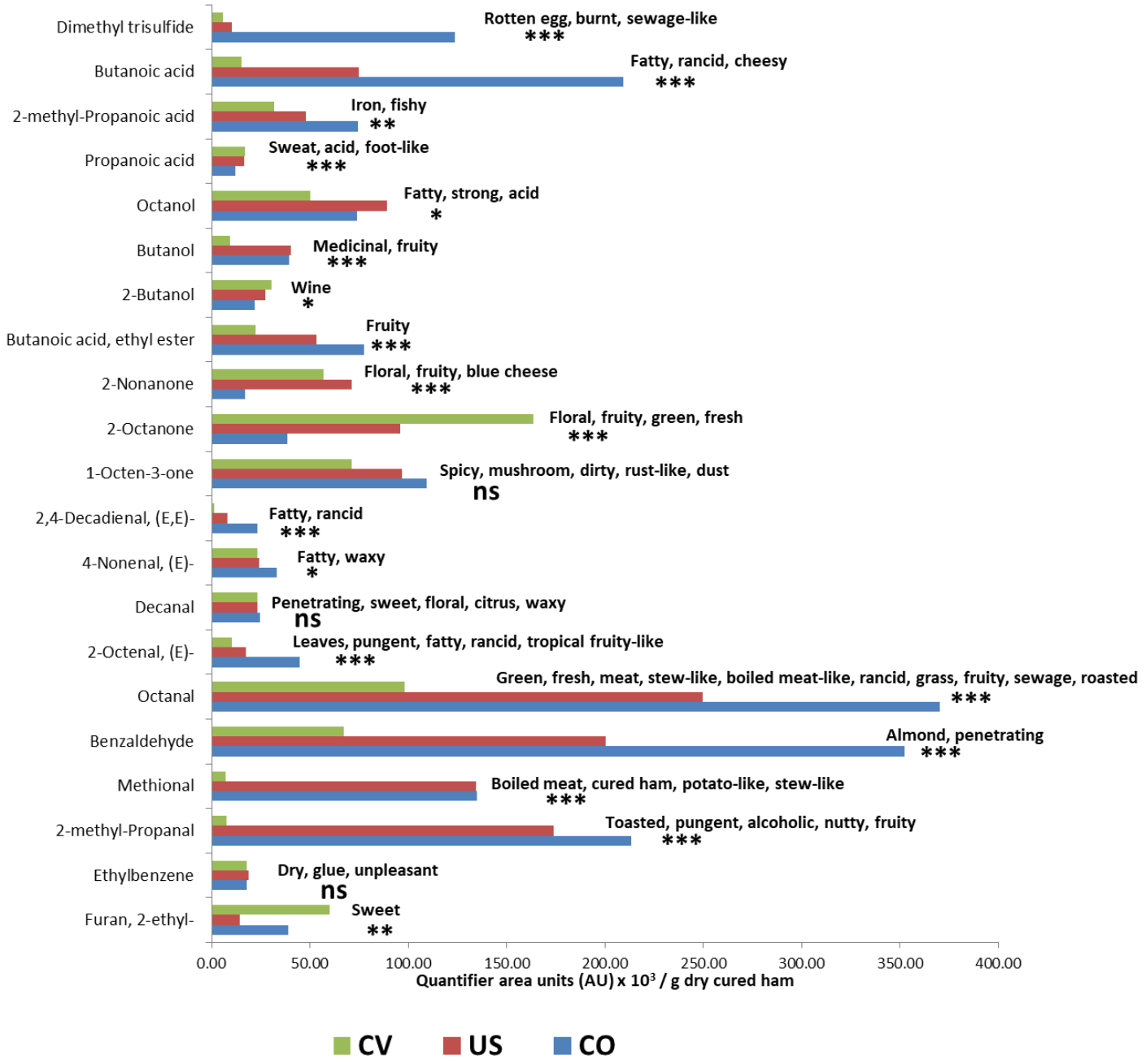

Dimethyl trisulfide
Rotten egg, burnt, sewage-like
***
Butanoic acid
Fatty, rancid, cheesy
***
2-methyl-Propanoic acid
Iron, fishy
**
Propanoic acid
Sweat, acid, foot-like
***
Octanol
Fatty, strong, acid
*
Butanol
Medicinal, fruity
***
2-Butanol
Wine
*
Butanoic acid, ethyl ester
Fruity
***
2-Nonanone
Floral, fruity, blue cheese
***
2-Octanone
Floral, fruity, green, fresh
***
1-Octen-3-one
Spicy, mushroom, dirty, rust-like, dust
ns
2,4-Decadienal, (E,E)-
Fatty, rancid
***
4-Nonenal, (E)-
Fatty, waxy
*
Decanal
Penetrating, sweet, floral, citrus, waxy
ns
2-Octenal, (E)-
Leaves, pungent, fatty, rancid, tropical fruity-like
***
Octanal
Green, fresh, meat, stew-like, boiled meat-like, rancid, grass, fruity, sewage, roasted
***
Benzaldehyde
Almond, penetrating
***
Methional
Boiled meat, cured ham, potato-like, stew-like
***
2-methyl-Propanal
Toasted, pungent, alcoholic, nutty, fruity
***
Ethylbenzene
Dry, glue, unpleasant
ns
Furan, 2-ethyl-
Sweet
**
0.00
50.00
100.00
150.00
200.00
250.00
300.00
350.00
400.00
Quantifier area units (AU) x 10³ / g dry cured ham
CV
US
CO

Figure 3

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.

Highlights:

- Temperature and ultrasound were essayed for decrease adhesiveness in ham.
- The effect of these treatments on free amino acid and volatile contents was studied.
- Temperature and ultrasound significantly decreased the adhesiveness of hams.
- Total free amino acid content significantly increased after both treatments.
- Temperature and ultrasound significantly decreased the total volatile content.

Table 1. Effect of treatments on free amino acids content (expressed as mg/100 g dry matter) in dry-cured ham. Values are means of the results from twenty-six samples of each group

	Tratamiento			SEM	*p-value*
	CO	**US**	**CV**		
Aspartic acid	164.65[a]	212.10[b]	149.32[a]	5.122	<0.001
Serine	191.48[a]	243.71[b]	204.82[a]	5.820	<0.001
Glutamic acid	430.61[a]	544.77[b]	463.93[a]	12.375	<0.001
Glycine	187.99[a]	245.58[c]	216.85[b]	5.917	<0.001
Histidine	99.02[a]	133.55[b]	113.51[a]	3.641	<0.001
Taurine	80.95[a]	102.75[b]	100.04[b]	2.592	<0.001
Arginine	364.86[a]	518.93[b]	361.99[a]	14.676	<0.001
Threonine	218.46[a]	281.96[c]	250.30[b]	6.642	<0.001
Alanine	398.16[a]	544.41[c]	461.75[b]	12.949	<0.001
Proline	287.99[a]	372.34[c]	330.99[b]	8.804	<0.001
Cisteine	287.14[a]	437.18[b]	417.09[b]	17.045	<0.001
Tyrosine	181.33[a]	228.49[b]	219.62[b]	6.942	<0.001
Valine	385.79[a]	484.95[b]	428.48[a]	10.053	<0.001
Metionine	213.90[a]	259.31[b]	250.63[b]	6.074	<0.001
Lysine	247.69[a]	351.95[b]	276.72[a]	9.506	<0.001
Isoleucine	364.94[a]	411.06[b]	421.89[b]	8.196	<0.001
Leucine	608.59[a]	750.85[b]	700.38[b]	15.831	<0.001
Phenilalanine	391.01[a]	495.85[b]	459.91[b]	11.808	<0.001
Total Aas	**5278.18[a]**	**6691.53[b]**	**6067.45[b]**	**148.807**	**<0.001**
Sweet[1]	**1328.43[a]**	**1705.69[c]**	**1499.88[b]**	**33.752**	**<0.001**
Bitter[2]	**2014.89[a]**	**2289.93[b]**	**2256.99[b]**	**36.002**	**<0.001**
Acid[3]	**699.95[a]**	**904.94[b]**	**765.60[a]**	**16.902**	**<0.001**
Aged[4]	**601.69[a]**	**767.19[b]**	**645.23[a]**	**14.888**	**<0.001**

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

SEM: standard error of mean.

Treatments: CO= control (without treatment), CV= conventional thermal treatments and US= thermal treatment assisted by power ultrasound

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

Table 2

Effect of treatments on volatile compounds content (expressed as quantifier area units (AU) x 10^3 / g dry cured ham. Values are means of the results from twenty-six samples of each group

Compound	*m/z*	LRI	R	Treatment			SEM	*P-value*
				CO	US	CV		
Pentane	43	500	*ms, lri, s*	883.71[a]	688.22[a]	1471.54[b]	94.956	0.005
Pentane, 2-methyl-	71	543	*ms, lri*	2.57[a]	3.29[ab]	4.50[b]	0.289	0.023
1-Butene, 2,3-dimethyl-	57	571	*ms*	19.51[a]	10.68[a]	30.18[b]	1.734	<0.001
n-Hexane	69	600	*ms, lri, s*	810.40[b]	529.80[a]	1541.71[c]	61.771	<0.001
Heptane	71	700	*ms, lri, s*	802.78	514.56	879.78	68.817	0.103
Pentane, 2,3,4-trimethyl-	71	756	*ms, lri*	232.76[a]	365.58[ab]	437.24[b]	26.540	0.003
Pentane, 2,3,3-trimethyl-	71	763	*ms, lri*	319.34[a]	508.02[b]	620.06[b]	34.305	<0.001
Pentane, 3-ethyl-	70	770	*ms, lri*	51.97[a]	77.48[ab]	85.39[b]	5.219	0.015
1-Pentene, 3-ethyl-2-methyl-	83	774	*ms*	32.98	37.73	45.65	2.220	0.069
Hexane, 2,2,5-trimethyl-	57	799	*ms*	374.97[a]	655.05[ab]	705.58[b]	51.550	0.010
Octane	85	800	*ms, lri, s*	1942.31	1335.15	1731.67	154.326	0.257
2-Octene, (E)-	112	833	*ms, lri*	201.22	122.73	157.6	14.935	0.078
Heptane, 3,4,5-trimethyl-	85	842	*ms*	67.19[a]	110.46[b]	120.25[b]	7.106	0.002
3-Octene, (E)-	112	845	*ms, lri*	84.68	59.41	70.66	6.160	0.217
Octane, 2-methyl-	71	899	*ms*	12.42	15.12	13.79	1.002	0.530
Hexane, 2,2,5,5-tetramethyl-	57	914	*ms, lri*	301.96	409.36	394.91	26.669	0.168
4-Nonene	70	926	*ms*	130.55	148.11	173.08	7.236	0.057
Nonane	126	900	*ms, lri, s*	131.63[a]	167.86[ab]	193.45[b]	9.614	0.024
Heptane, 2-methyl-3-methylene-	57	930	*ms*	12.74[a]	14.51[ab]	17.80[b]	0.743	0.020
2-Octene, 4-ethyl-	69	982	*ms*	121.06	109.24	139.94	7.447	0.322
Octane, 3-methyl-6-methylene-	70	985	*ms*	204.18[a]	223.88[ab]	286.28[b]	12.678	0.028
Octane, 4-ethyl-	69	991	*ms*	72.43[a]	83.39[ab]	99.48[b]	4.114	0.026
Heptane, 3,3,4-trimethyl-	69	994	*ms*	6.01[a]	11.98[b]	3.49[a]	0.730	<0.001
Pentane, 3,3-dimethyl-	85	995	*ms*	6.14	5.74	7.14	0.432	0.483
Decane	57	1000	*ms, lri, s*	392.40	484.05	448.96	35.082	0.536
Nonane, 2,3-dimethyl-	71	1003	*ms*	62.32	61.17	73.08	3.761	0.440
1-Octene, 2,6-dimethyl-	56	1010	*ms*	72.47	78.95	89.54	4.118	0.252
3-Octene, 4-ethyl-	69	1012	*ms*	23.62	22.29	26.35	1.302	0.519
Nonane, 3-methylene-	70	1022	*ms*	165.31	193.91	219.60	9.675	0.068
Heptane, 2,2,4,6,6-pentamethyl-	57	1027	*ms, lri*	3130.36[ab]	6386.68[b]	2772.86[a]	571.676	0.023
3-Ethyl-3-hexene	83	1042	*ms*	46.18[a]	68.29[a]	99.93[b]	5.404	<0.001
Undecane, 3,6-dimethyl-	57	1068	*ms*	247.95[ab]	333.34[b]	119.46[a]	31.537	0.042
Tridecane, 6-methyl-	57	1079	*ms, lri*	241.55	296.61	296.67	18.192	0.326
Undecane, 2,5-dimethyl-	57	1085	*ms*	159.26	140.65	150.96	11.186	0.788
Decane, 2,3,5-trimethyl-	57	1099	*ms*	102.23[b]	56.83[a]	81.27[ab]	7.435	0.032
Undecane	57	1100	*ms, lri, s*	930.86	1346.47	1216.44	83.082	0.085
2,3-Dimethyl-3-heptene, (Z)-	83	1123	*ms, lri*	56.04[b]	25.71[a]	10.65[a]	4.093	<0.001
2-Undecene, 9-methyl-, (Z)-	70	1132	*ms*	368.85	345.35	367.91	22.501	0.900
5-Undecene, 6-methyl-	168	1144	*ms*	11.24	8.17	9.33	0.741	0.202
4,4-Dipropylheptane	85	1153	*ms*	51.23	43.30	50.12	3.096	0.548
2-Undecene, 3-methyl-, (E)-	70	1181	*ms*	60.96	55.41	61.11	3.488	0.774
4-Nonene, 5-butyl-	70	1197	*ms*	24.26	23.38	20.87	1.532	0.678
Dodecane	57	1200	*ms, lri, s*	664.51	948.13	849.77	53.501	0.066
Decane, 3-ethyl-3-methyl-	57	1228	*ms*	50.22	42.58	46.32	2.933	0.551
Dodecane, 2-methyl-	57	1233	*ms*	23.00[a]	38.36[b]	30.39[ab]	2.057	0.005
1-Tetradecene	97	1236	*ms, lri*	31.84	30.42	28.93	2.097	0.857
Tridecane	71	1300	*ms, lri, s*	228.76	318.27	217.88	21.114	0.131
Tridecane, 3-methyl-	85	1304	*ms*	31.82	38.27	37.84	1.868	0.252
Total Aliphatic hydrocarbons				**15578.28**	**19062.05**	**17144.10**	**1014.413**	**0.356**
Furan, 2-ethyl-	81	703	*ms, lri*	38.75[ab]	14.06[a]	60.00[b]	4.756	0.001
Toluene	92	804	*ms*	122.47[a]	131.23[a]	178.32[b]	5.716	<0.001
Cyclobutane, 1,1,2,3,3-pentamethyl-	70	813	*ms*	247.78	268.52	288.93	13.907	0.490
Ethylbenzene	91	917	*ms, lri*	17.64	18.84	17.70	0.814	0.811

Benzene, 1,3-dimethyl-	106	926	*ms*	19.44	21.44	21.39	0.603	0.267
2-n-Butyl furan	81	944	*ms, lri*	35.70	32.04	42.78	2.845	0.383
Cyclopentane, 1-ethyl-3-methyl-	83	1123	*ms*	56.04^{b}	25.71^{a}	10.65^{a}	4.093	<0.001
Cyclopentane, ethyl-	98	1148	*ms, lri*	300.84^{c}	173.68^{b}	38.57^{a}	20.284	<0.001
Total Aromatic and cyclic hycrocarbons				**808.45**	**743.01**	**769.51**	**26.041**	**0.565**
Total Hydrocarbons				**16867.18**	**19912.67**	**17932.30**	**1045.388**	**0.479**
Propanal	58	526	*ms, lri, s*	139.01^{a}	102.85^{a}	751.47^{b}	43.600	<0.001
Propanal, 2-methyl-	72	557	*ms, lri*	213.22^{b}	173.69^{b}	7.43^{a}	16.502	<0.001
Butanal	72	584	*ms, lri, s*	23.16^{c}	10.81^{b}	1.45^{a}	1.688	<0.001
Butanal, 3-methyl-	58	659	*ms, lri*	1968.06^{c}	1240.06^{b}	68.91^{a}	142.214	<0.001
Butanal, 2-methyl-	57	671	*ms, lri*	1139.71^{b}	929.14^{b}	43.06^{a}	84.003	<0.001
Pentanal	57	728	*ms, lri, s*	951.76	640.68	697.89	65.639	0.090
2-Butenal, 2-methyl-	84	801	*ms*	104.37^{b}	55.38^{a}	27.29^{a}	7.598	<0.001
Hexanal	56	865	*ms, lri, s*	12264.83^{c}	5747.78^{b}	185.13^{a}	889.713	<0.001
Heptanal	70	974	*ms, lri, s*	853.54^{c}	401.98^{b}	25.49^{a}	68.206	<0.001
Methional	104	999	*ms, lri*	134.75^{b}	134.52^{b}	7.04^{a}	12.331	<0.001
Benzaldehyde	106	1045	*ms, lri*	352.12^{c}	200.47^{b}	67.03^{a}	22.052	<0.001
Octanal	56	1066	*ms, lri, s*	370.02^{c}	249.58^{b}	98.19^{a}	23.992	<0.001
5-Ethylcyclopent-1-enecarboxaldehyde	124	1099	*ms*	32.99^{b}	17.82^{a}	10.03^{a}	2.308	<0.001
Benzeneacetaldehyde	91	1119	*ms, lri*	796.26^{c}	356.03^{b}	37.78^{a}	52.710	<0.001
2-Octenal, (E)-	70	1123	*ms, lri*	44.78^{b}	17.22^{a}	10.22^{a}	3.112	<0.001
Decanal	81	1129	*ms, lri, s*	24.68	23.26	23.18	1.663	0.912
Nonanal	57	1148	*ms, lri, s*	614.70^{c}	380.07^{b}	133.97^{a}	38.155	<0.001
4-Nonenal, (E)-	83	1201	*ms*	33.21^{b}	23.96^{ab}	23.29^{a}	1.657	0.013
Benzaldehyde, 3-ethyl-	134	1209	*ms*	33.46^{b}	27.15^{b}	8.76^{a}	2.527	<0.001
2-Decenal, (E)-	70	1272	*ms, lri*	28.90^{b}	19.66^{ab}	13.75^{a}	1.793	0.001
2,4-Decadienal, (E,E)-	81	1315	*ms, lri*	23.10^{b}	8.08^{a}	1.22^{a}	2.199	<0.001
2-Undecenal	95	1339	*ms, lri*	6.56^{b}	2.44^{a}	2.76^{a}	0.624	0.004
Pentadecanal-	82	1516	*ms, lri, s*	3.90^{a}	9.02^{b}	4.73^{a}	0.682	0.003
Total Aldehyde				**23509.08^{c}**	**10307.72^{b}**	**2381.68^{a}**	**1562.858**	**<0.001**
Acetone	58	528	*ms*	246.04^{a}	438.13^{b}	958.64^{c}	50.416	<0.001
2,3-Hexanedione	41	562	*ms*	391.05^{b}	226.53^{a}	696.97^{c}	30.694	<0.001
2-Butanone	72	596	*ms*	177.17^{a}	264.28^{b}	504.65^{c}	22.630	<0.001
Cyclopentanone, 3-methyl-	56	667	*ms*	30.74^{ab}	18.76^{a}	34.05^{b}	2.459	0.043
2-Pentanone	86	720	*ms, lri*	101.75^{a}	78.17^{a}	305.68^{b}	25.871	0.001
Acetoin	45	787	*ms, lri*	484.13^{a}	501.60^{a}	2031.51^{b}	153.676	<0.001
3-Heptanone	57	960	*ms, lri*	43.80	37.03	37.54	1.883	0.225
2-Heptanone	58	967	*ms, lri*	427.95^{a}	664.14^{ab}	980.43^{b}	62.048	0.001
Cyclohexanone, 2-ethyl-	69	972	*ms*	39.00^{a}	42.78^{a}	65.73^{b}	3.247	0.002
2-Nonen-4-one	69	979	*ms*	13.48	14.36	17.24	0.940	0.272
2-Hepten-4-one, 6-methyl-	69	992	*ms*	72.65^{a}	80.61^{ab}	99.82^{b}	3.864	0.015
4-Octanone, 5-hydroxy-2,7-dimethyl-	69	1042	*ms*	9.29^{a}	18.03^{ab}	21.64^{b}	1.615	0.003
1-Octen-3-one	70	1046	*ms, lri*	109.18	96.80	71.31	8.502	0.202
5-Hepten-2-one, 6-methyl-	69	1056	*ms, lri*	104.35^{ab}	93.37^{a}	134.10^{b}	5.814	0.026
2-Octanone	58	1059	*ms, lri*	38.35^{a}	95.71^{a}	163.52^{b}	12.653	<0.001
3-Nonanone	113	1134	*ms*	23.48	21.34	23.80	1.588	0.818
1-Hexanone, 5-methyl-1-phenyl-	105	1137	*ms*	15.19^{a}	28.98^{b}	24.08^{b}	1.564	<0.001
2-Nonanone	58	1141	*ms, lri*	16.85^{a}	71.11^{b}	56.62^{b}	6.375	<0.001
2(3H)-Furanone, 5-ethyldihydro-	85	1158	*ms, lri*	187.86	226.67	199.86	8.500	0.156
5-Hexen-3-one	57	1161	*ms*	48.92	38.56	53.49	3.652	0.298
2,6-Bis(1,1-dimethylethyl)-4-(1-oxopropyl)phenol	233	1448	*ms*	11.04^{b}	0.00^{a}	0.00^{a}	1.497	0.001
Total Ketone				**2322.78^{a}**	**3046.03^{b}**	**6772.32^{c}**	**265.182**	**<0.001**
Acetic acid ethenyl ester	86	588	*ms*	25.62^{a}	17.51^{a}	50.61^{b}	3.166	<0.001
Ethyl Acetate	61	598	*ms*	107.45	162.28	142.48	13.452	0.210
Methane, oxybis[dichloro-	83	611	*ms*	224.46	251.18	231.85	14.170	0.734
Propanoic acid, ethyl ester	57	737	*ms*	46.38^{b}	15.79^{a}	19.06^{a}	3.404	<0.001
Butanoic acid, ethyl ester	71	855	*ms*	77.53^{c}	53.05^{b}	22.14^{a}	4.569	<0.001
Butanoic acid, 2-methyl-, ethyl ester	102	908	*ms*	46.49	49.14	39.04	3.892	0.624

Butanoic acid, 3-methyl-, ethyl ester	88	913	*ms*	121.86^{ab}	138.61^{b}	67.83^{a}	10.093	0.024
Oxalic acid, butyl propyl ester	57	936	*ms*	131.63^{a}	167.86^{ab}	193.45^{b}	9.614	0.024
Ethanol, 2-butoxy-	57	985	*ms, lri*	394.15^{b}	296.66^{ab}	218.86^{a}	22.783	0.004
Carbonic acid, bis(2-ethylhexyl) ester	112	1003	*ms*	25.20	25.06	28.09	1.605	0.736
Hexanoic acid, ethyl ester	88	1050	*ms*	184.39^{b}	150.70^{b}	79.11^{a}	11.285	<0.001
2-Piperidinecarboxylic acid, 1-acetyl-, ethyl ester	84	1124	*ms*	30.54^{b}	18.80^{a}	15.15^{a}	1.887	0.001
Carbonic acid, tridecyl vinyl ester	57	1168	*ms*	210.11^{a}	163.66^{a}	189.81^{a}	15.263	0.447
Octanoic acid, ethyl ester	88	1204	*ms*	75.26^{b}	77.21^{b}	42.04^{a}	4.187	0.001
Decanoic acid, ethyl ester	88	1336	*ms*	33.57^{b}	27.32^{b}	12.77^{a}	2.519	0.002
2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	71	1442	*ms*	3.42^{a}	3.40^{a}	2.43^{a}	0.182	0.064
Total Esther and ether				**1906.99^{b}**	**1680.82^{ab}**	**1385.33^{a}**	**68.273**	**0.006**
Isopropyl Alcohol	45	532	*ms*	119.01^{ab}	163.82^{b}	100.93^{a}	9.654	0.039
1-Propanol	59	572	*ms*	39.39^{ab}	59.98^{b}	23.41^{a}	3.963	0.002
2-Butanol	45	607	*ms, lri*	21.64	27.36	30.26	1.483	0.043
1-Butanol	56	707	*ms, lri*	39.26^{b}	40.08^{b}	9.13^{a}	3.127	<0.001
1-Penten-3-ol	57	730	*ms*	853.31	621.14	784.02	47.894	0.122
2-Pentanol	45	751	*ms*	124.97	209.61	202.82	18.563	0.088
1-Butanol, 3-methyl-	55	808	*ms, lri*	239.69^{a}	1169.80^{b}	3556.89^{c}	253.843	<0.001
1-Butanol, 2-methyl-	57	812	*ms*	39.06^{a}	238.09^{b}	581.42^{c}	42.813	<0.001
1-Pentanol	55	847	*ms, lri*	576.25^{b}	299.13^{a}	189.49^{a}	43.802	<0.001
2-Propanol, 2-methyl-	59	894	*ms*	22.58^{b}	9.71^{a}	17.36^{ab}	1.924	0.016
2,3-Butanediol, [S-(R*,R*)]-	45	909	*ms*	69.08^{b}	8.56^{a}	2.13^{a}	7.003	<0.001
3-Pentanol, 2,4-dimethyl-	73	954	*ms*	13.50	18.68	24.18	2.149	0.129
1-Heptanol	70	1046	*ms*	109.18	96.80	71.31	8.502	0.202
1-Octen-3-ol	57	1051	*ms, lri*	3543.17	3818.07	3922.68	236.699	0.789
1-Heptanol, 2,4-diethyl-	69	1085	*ms*	112.27	71.78	77.41	9.031	0.108
2-Ethyl-1-hexanol	57	1094	*ms*	11.36^{ab}	10.53^{a}	15.90^{b}	0.875	0.048
4-Ethylcyclohexanol	81	1104	*ms*	90.23^{a}	129.55^{ab}	141.39^{b}	8.253	0.019
Benzyl alcohol	108	1124	*ms, lri*	131.16	145.59	153.53	7.361	0.444
1-Octanol	56	1127	*ms, lri*	73.90^{ab}	88.89^{b}	49.90^{a}	5.781	0.043
4-Methyl-5-decanol	55	1162	*ms*	25.30^{a}	36.53^{a}	74.05^{b}	5.088	<0.001
p-Cresol	107	1178	*ms*	30.50	31.28	28.20	1.333	0.687
Phenylethyl Alcohol	92	1182	*ms*	13.89^{a}	186.88^{a}	883.92^{b}	65.261	<0.001
1-Tetradecanol	68	1225	*ms*	28.08	31.26	33.29	1.363	0.281
1,4-Benzenediol, 2,5-bis(1,1-dimethylethyl)-	222	1485	*ms*	0.27^{a}	0.41^{b}	0.27^{a}	0.017	<0.001
Total Alcohol				**6548.61^{a}**	**8599.43^{a}**	**12199.24^{b}**	**487.720**	**<0.001**
Propanoic acid	74	827	*ms, lri*	12.07	16.39	16.71	2.193	0.606
Propanoic acid, 2-methyl-	73	888	*ms, lri*	74.38^{b}	47.64^{ab}	31.63^{a}	5.693	0.005
Butanoic acid	60	918	*ms, lri*	209.13^{c}	74.58^{b}	15.13^{a}	14.471	<0.001
Butanoic acid, 3-methyl-	60	969	*ms, lri*	427.98	329.99	366.87	33.667	0.459
Pentanoic acid	60	1083	*ms, lri*	428.30^{c}	274.79^{b}	7.68^{a}	28.766	<0.001
Octanoic acid	60	1224	*ms*	36.67^{c}	20.14^{b}	4.08^{a}	2.717	<0.001
Total Carboxylic acid				**1172.40^{c}**	**950.08^{b}**	**316.57^{a}**	**58.148**	**<0.001**
Fumaronitrile	78	646	*ms*	27.19^{b}	17.32^{a}	23.53^{ab}	1.418	0.011
3-(1'-pyrrolidinyl)-2-butanone	98	906	*ms*	92.62	95.73	121.88	5.438	0.078
Pyrazine, 2,6-dimethyl-	108	978	*ms, lri*	347.01^{a}	337.27^{a}	478.72^{b}	14.720	<0.001
1-(1'-pyrrolidinyl)-2-butanone	84	982	*ms*	90.39	97.20	117.94	5.324	0.110
Total Nitrogenous compounds				**561.37^{a}**	**550.57^{a}**	**747.76^{b}**	**20.616**	**<0.001**
Carbon disulfide	76	533	*ms*	157.74^{b}	77.69^{a}	195.02^{b}	11.366	<0.001
Disulfide, dimethyl	94	781	*ms, lri*	1740.04^{b}	206.48^{a}	738.87^{a}	141.238	<0.001
Dimethyl trisulfide	126	1035	*ms, lri*	123.40^{b}	10.27^{a}	5.82^{a}	10.579	<0.001
Sulfurous acid, decyl hexyl ester	85	1156	*ms*	110.15	122.77	104.36	11.499	0.835
Sulfurous acid, butyl dodecyl ester	85	1304	*ms*	31.82	38.24	37.81	1.862	0.254
Total Sulfur compounds				**2213.62^{b}**	**443.46^{a}**	**1081.88^{a}**	**161.357**	**<0.001**
Total Compounds				**56662.84^{b}**	**45848.47^{a}**	**48407.25^{ab}**	**1697.399**	**0.013**

[a-c] Mean values in the same row (corresponding to the same parameter) 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, 1.4 µm 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, & Dominguez, 2014; Lorenzo, & Carballo, 2015; Pateiro, Franco, Carril, & Lorenzo, 2015; Pérez-Santaescolástica *et al*., 2018; Purriños et al., 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), CV= conventional thermal treatments and US= thermal treatment assisted by power ultrasound