# Volatile Compounds, Odor, and Aroma of La Serena Cheese High-Pressure Treated at Two Different Stages of Ripening

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### ABSTRACT

La Serena cheeses made from raw Merino ewe's milk were high-pressure (HP) treated at 300 or 400 MPa for 10 min on d 2 or 50 after manufacture. Ripening of HPtreated and control cheeses proceeded until d 60 at 8°C. Volatile compounds were determined throughout ripening, and analysis of related sensory characteristics was carried out on ripe cheeses. High-pressure treatments on d 2 enhanced the formation of branched-chain aldehydes and of 2-alcohols except 2-butanol, but retarded that of n-aldehydes, 2-methyl ketones, dihydroxy-ketones, n-alcohols, unsaturated alcohols, ethyl esters, propyl esters, and branched-chain esters. Differences between HP-treated and control cheeses in the levels of some volatile compounds tended to disappear during ripening. The odor of ripe cheeses was scarcely affected by HP treatments on d 2, but aroma quality and intensity scores were lowered in comparison with control cheese of the same age. On the other hand, HP treatments on d 50 did not influence either the volatile compound profile or the sensory characteristics of 60d-old cheese.

**Key words:** high pressure, La Serena cheese, volatile compounds, aroma

# INTRODUCTION

High-pressure (**HP**) treatment is an adequate procedure to eliminate undesirable microorganisms in foods that might be altered by thermal processes. Moderate HP treatments have been successfully applied for cheese production (O'Reilly et al., 2001; Trujillo et al., 2002). Most of the research carried out on HP treatment of cheese is related to increasing its safety and shelf life through the inactivation of pathogenic and spoilage microorganisms (Capellas et al., 1996; Gallot-Lavallée, 1998; O'Reilly et al., 2000a; Carminati et al., 2004; Arqués et al., 2005; Rodríguez et al., 2005). The effect of HP treatments on the acceleration of proteolysis during ripening of some cheese varieties has also been investigated, with variable results depending on treatment conditions, cheese variety, age of cheese at treatment, and analytical methods used (Yokoyama et al., 1992; O'Reilly et al., 2000b; Saldo et al., 2001, 2002; Juan et al., 2004; Ávila et al., 2006b).

More than 600 volatile compounds have been identified in different cheese varieties (Molimard and Spinnler, 1996; Fox and Wallace, 1997; Curioni and Bosset, 2002). Many of these compounds have been related to particular odor and aroma notes, and are responsible for cheese sensory characteristics (Urbach, 1993). Information available on the effect of HP treatment on cheese volatile compounds is scarce. Published works have dealt with the effects on the volatile profile of Garrotxa cheese made from pasteurized goat's milk (Saldo et al., 2003), Hispánico cheese made from a mixture of pasteurized cow's and ewe's milks (Ávila et al., 2006a), and a semihard cheese made from pasteurized ewe's milk (Juan et al., 2007). To our knowledge, the effect of HP treatment on the volatile compounds of raw milk cheese has not been investigated.

La Serena cheese is a Spanish variety made from raw Merino ewe's milk, without added starter cultures and coagulated with an extract of Cynara cardunculus (thistle) flowers. It is consumed after ripening for a minimum of 60 d. Its volatile fraction is very rich in alcohols and esters, and its odor and aroma have lactic, fruity-flowery, and animal notes. In particular, ethyl esters and some alcohols seem to play a key role in the aroma of La Serena cheese (Carbonell et al., 2002). The high pH values of this cheese during the first stages of ripening, together with its high moisture and low salt content, are favorable for the growth of contaminating microorganisms, including pathogens (Fernández del Pozo et al., 1988; Sánchez-Rey et al., 1993). In previous works, the effects of HP treatments on the undesirable microorganisms present in 2 batches of La Serena cheese (Arqués et al., 2006) and on their proteolysis and texture (Garde et al., 2007) were studied. The objective of the present work was to investigate the effect of HP treatments on the volatile compounds and related

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sensory characteristics (odor and aroma) of the same batches of La Serena cheese to complete the validation of the process.

# MATERIALS AND METHODS

#### Cheese Manufacture

La Serena cheeses studied in the present work were those described in previous papers (Arqués et al., 2006; Garde et al., 2007). Two batches were made, each from 400 L of refrigerated, raw Merino ewe's milk with no added starter cultures, at the same dairy on different days. Milk was coagulated at 30°C for 75 min with an aqueous extract of macerated *C. cardunculus* flowers. Curds were cut into 20-mm cubes, held at 30°C for 15 min, and distributed into cylindrical molds. Cheeses, 18 cm in diameter and 6 cm high, were pressed for 6 h and salted by rubbing dry salt twice on the surface. They were ripened at the dairy for 60 d at 8°C and 90% relative humidity.

# **HP Treatments**

Six cheeses from each batch were vacuum packed in CN300 bags (Cryovac Grace S. A., Barcelona, Spain) on d 2 and HP treated, 3 at 300 MPa and 3 at 400 MPa, for 10 min at an initial temperature of 10°C, by means of a 100-L-capacity discontinuous isostatic press at NC Hyperbaric (Burgos, Spain). Come-up times to reach 300 and 400 MPa were 4.5 and 5.9 min, respectively, and depressurization times were 1.4 and 1.8 min, respectively. The temperature of the water used as pressure-transmitting fluid remained below 14°C during the process. On d 50, 2 other cheeses from each batch were vacuum packed in CN300 bags and HP treated, one at 300 MPa and one at 400 MPa, for 10 min. After the HP treatments, cheeses were unpacked and allowed to ripen until d 60 after manufacture at the dairy with the rest of the batch. Nonpressurized cheeses from each batch served as controls. Cheeses were transported at 4°C to the laboratory at 3, 30, or 60 d of ripening for analysis.

### Analysis of Volatile Compounds

Cheese pieces, without rind, were wrapped in aluminum foil, vacuum packed, and held at -40°C until analysis. Prior to volatile extraction, frozen pieces were thawed overnight at 4°C. An automatic dynamic headspace apparatus (Purge and Trap, HP 7695, Hewlett-Packard, Palo Alto, CA) connected to a gas chromatograph-mass spectrometer (HP 6890-MSD HP 5973, Hewlett-Packard) was used for the analysis of volatile compounds. Duplicate 10-g cheese samples were homogenized in an analytical grinder (IKA, Labortechnik, Staufen, Germany), with 20 g of Na<sub>2</sub>SO<sub>4</sub> and 50  $\mu$ L of an aqueous solution containing 0.5 mg/mL of cyclohexanone [internal standard (**IS**)<sub>1</sub>] and camphor (IS<sub>2</sub>) as internal standards. An aliquot (2 g) of the mixture was subjected to helium purge in a 25-mL glass sparger (Schmidlin Co., Neuheim, Switzerland) at 50°C for 15 min, with 10 min of previous equilibrium. The analytical procedure and the identification and relative quantification of volatile compounds were carried out as previously described (Carbonell et al., 2002) by using selected ions. Relative abundances of compounds were expressed as percentages of their peak areas on the cyclohexanone peak area.

### Sensory Evaluation

Twelve trained panelists tasted the cheeses at 60 d of ripening. Panelists were presented with a representative slice of 3 cheeses per session, the control cheese and cheeses treated at 300 or 400 MPa on d 2, or the control cheese and cheeses treated at 300 or 400 MPa on d 50. Panelists were asked to score the quality and intensity of odor and aroma on a 0- to 10-point scale for each of these characteristics by using a horizontal line anchored at the middle, at the left end (lowest = 0points), and at the right end (highest = 10 points). Odor was defined as the olfactory sensation felt directly by the nose. Aroma was defined as the olfactory sensation felt retronasally upon mastication. A descriptive test was developed for La Serena cheese based on the guidelines of Berodier et al. (1997). Panelists were also asked to give a score, on a 0- to 10-point scale, to the following families of odor and aroma descriptors: "lactic," "vegetable," "floral," "toasted," and "animal."

### Statistical Analysis

Statistics were performed by means of the SPSS Win 8.0 software package (SPSS Inc., Chicago, IL). Analysis of variance was carried out on analytical variables, with HP treatment, days of ripening, and cheese-making experiment as the main effects. Comparison of means was performed with Tukey's test. A principal components analysis (**PCA**) with Varimax rotation was carried out on selected volatile compounds and sensory attributes (odor and aroma quality, odor and aroma intensity, and families of descriptors) of 60-d-old HP-treated and control cheeses.

#### RESULTS

# Volatile Compounds

Purge and trap extraction coupled with GC-MS allowed the identification of 60 volatile compounds (8

aldehydes, 6 ketones, 19 alcohols, 3 carboxylic acids, 13 esters, 4 terpenes, 5 hydrocarbons, and 2 aromatic compounds) in the headspace of La Serena cheeses. Six volatile compounds (heptanal, nonanal, decanal, 1-heptanol, toluene, and ethyl benzene) were not significantly influenced (P > 0.05) by HP treatment or by cheese age.

Thirteen volatile compounds (hexanal, 2-heptanone, 1-pentanol, 1-octanol, 2-methyl-2-propanol, hexanoic acid, heptane, octane, 3,7-dimethyl-2-octene, and the 4 terpenes) were significantly (P < 0.05) influenced by cheese age, but not by HP treatment. The levels of these volatile compounds, except those of hexanal, increased with cheese age.

High-pressure treatments on d 2 had significant (P< 0.05) effects on the levels of 41 volatile compounds, 36 of which (acetaldehyde, 2-propenal, 2-butanone, 2pentanone, 2,3-butanedione, 3-hydroxy-2-butanone, ethanol, 1-propanol, 1-butanol, 1-hexanol, 2-butoxyethanol, 2-butanol, 2-pentanol, 2-heptanol, 2-methyl-1propanol, 3-methyl-1-butanol, the 4 unsaturated alcohols, acetic acid, butanoic acid, the 13 esters, and hexane) increased significantly (P < 0.05) with cheese age, one (3-methylbutanal) decreased, and the rest (2-methylpropanal, 2-propanone, 2-propanol, and pentane) were not significantly influenced by cheese age. Cheese treatment by HP on d 50 had a significant (P < 0.05) effect on the levels of only 6 volatile compounds (2,3butanedione, 3-hydroxy-2-butanone, 3-methyl-3-buten-1-ol, butanoic acid, ethyl butanoate, and ethyl decanoate).

Aldehydes and Ketones. The relative abundance of aldehydes identified in the volatile fraction of La Serena cheeses is shown in Figure 1. On d 3, acetaldehyde was at significantly (P < 0.05) lower levels in cheeses HP treated on d 2 than in control cheese, but differences between cheeses were no longer significant on d 60. The relative abundance of 2-propenal was lower in cheeses HP treated on d 2 than in control cheese from d 30 onward, with the lowest level of 2-propenal on d 60 among all cheeses being that of cheese treated at 400 MPa on d 2. In contrast, the highest level of 3-methylbutanal on d 30 and 60 was found for cheese treated at 400 MPa on d 2. High-pressure treatment of cheeses on d 50 did not have a significant effect on the levels of any of the aldehydes present in 60-d-old cheeses.

Levels of ketones are shown in Figure 2. During the first month of ripening, the level of 2-propanone was significantly (P < 0.05) higher in cheese treated at 400 MPa on d 2 than in the other 2 cheeses, but this difference had disappeared by d 60. In contrast, HP treatments on d 2, in particular at 400 MPa, strongly inhibited the formation of 2-butanone during ripening and, to a lesser degree, inhibited the formation of other ke-

tones. Both HP treatments on d 50 influenced the levels of 2,3-butanedione and 3-hydroxy-2-butanone, which were significantly lower on d 60 in HP-treated cheeses than in control cheese.

Alcohols, Carboxylic Acids, and Esters. Alcohols (Figure 3) were quantitatively the main family of compounds in the volatile fraction of La Serena cheeses. The relative abundances of ethanol, 1-propanol, 1-butanol, 1-hexanol, 2-butoxyethanol, 2-butanol, 2-heptanol, 2-methyl-1-propanol, 3-methyl-1-butanol, 2-propen-1ol, and 2-buten-1-ol were significantly (P < 0.05) lower during the first month of ripening in cheeses HP treated on d 2 than in control cheese, but the differences were no longer significant by d 60 for ethanol, 2-heptanol, 2-methyl-1-propanol, and 3-methyl-1-butanol. On the contrary, cheeses HP treated on d 2 showed higher levels of 2-propanol, 3-methyl-3-buten-1-ol, and 3methyl-2-buten-1-ol than control cheese at the end of the ripening period. High-pressure treatments on d 50 did not have significant effects on the levels of alcohols in 60-d-old cheeses, with the exception of 3-methyl-3buten-1-ol, which was lower in cheese treated at 400 MPa than in control cheese or in cheese treated at 300 MPa.

The effect of HP treatments on carboxylic acids (Figure 4) did not follow a definite pattern. Cheese HP treated on d 2 at 400 MPa showed the lowest (P < 0.05) levels of acetic and butanoic acids on d 30. In contrast, cheese HP treated on d 2 at 300 MPa and cheese treated on d 50 at 400 MPa exhibited higher levels of butanoic acid than did control cheese on d 60.

Thirteen esters were identified in the volatile fraction of La Serena cheese (Figure 5). Ethyl esters, except for ethyl decanoate, propyl esters, and 1-methylpropyl acetate, were generally at lower levels in cheeses HP treated on d 2 than in control cheese. The differences between cheeses had disappeared by d 60 for ethyl acetate, ethyl propanoate, ethyl pentanoate, ethyl hexanoate, and ethyl octanoate. Ethyl decanoate was at higher levels in 60-d-old cheeses HP treated on d 2 than in the respective control cheese. Cheeses HP treated on d 50 showed lower levels of ethyl butanoate than did control cheese on d 60, whereas cheese HP treated on d 50 at 400 MPa showed higher levels of ethyl decanoate than did control cheese on d 60.

**Miscellaneous Compounds.** No significant differences between cheeses attributable to HP treatments were found in the case of terpenes, which had relative abundances in the ranges of 3.92 to 4.53 for  $\alpha$ -pinene, 0.19 to 0.26 for camphene, 0.27 to 0.30 for  $\delta$ -carene, and 0.34 to 0.38 for cymene on d 60 (data not shown). A significant effect of HP treatments was observed for pentane, hexane, and octane, with a tendency to show higher levels in HP-treated cheeses than in control

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Figure 1. Aldehydes in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean  $\pm$  SE of duplicate determinations in 2 experiments).

cheese (Figure 6), whereas heptane and 3,7-dimethyl-2-octene were not affected by HP treatments. Aromatic compounds, with relative abundances in the ranges of 1.64 to 2.21 for toluene and 0.10 to 0.14 for ethyl benzene on d 60 (data not shown), were not influenced by HP treatments.

Sensory Characteristics. The odor quality and intensity of 60-d-old La Serena cheese were not significantly (P < 0.05) influenced by HP treatments on d 2 (Table 1), but aroma quality and intensity were affected by treatment at 400 MPa on d 2, which resulted in significantly (P < 0.05) lower scores than those of control cheese. The effect of HP treatments on d 50 on the odor and aroma of 60-d-old La Serena cheese was negligible (Table 1). Scores for families of odor and aroma descriptors (lactic, vegetable, floral, toasted, and animal) were not significantly affected by any of the HP treatments applied on d 2 or 50.

# Correlation Between Volatile Compounds and Sensory Characteristics

A PCA was used to correlate volatile compounds with cheese sensory characteristics (Table 2 and Figure 7).



**Figure 2.** Ketones in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean ± SE of duplicate determinations in 2 experiments).

Principal component 1, which was strongly related to the intensity and quality of cheese aroma, explained 45.5% of the variance. Unsaturated alcohols, 2-butanol, propyl esters, 2-butanone, branched-chain esters, branched-chain alcohols, n-alcohols, 2-methyl ketones (not including 2-butanone), aroma intensity and quality, the aroma descriptor "animal," and odor quality correlated positively with principal component 1, whereas 2-alcohols (not including 2-butanol) and branched-chain aldehydes correlated negatively. Principal component 2, which was related to the aroma descriptors "vegetable" and "floral," explained 16.9% of the variance. Variables correlating positively with principal component 2 were n-alcohols, 2-butanone, aldehydes, propyl esters, 2-butanol, ethyl esters, the aroma descriptors "vegetable" and "floral," and odor intensity, whereas the aroma descriptor "lactic" correlated negatively.

Figure 8 shows the distribution of 60-d-old La Serena cheeses in the plane defined by principal components 1 and 2. Control cheese and cheeses HP treated on d 50 were separated by principal components 1 and 2 from cheeses HP treated on d 2.

### DISCUSSION

Volatile compounds found in the 2 batches of cheese analyzed in the present work totaled 60, a lower number than the 112 volatile compounds reported for La Serena cheese in a previous study carried out in our laboratory (Carbonell et al., 2002). Because the same analytical technique was used in both works, the richer diversity

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**Figure 3.** Alcohols in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean ± SE of duplicate determinations in 2 experiments).



**Figure 3 (Continued).** Alcohols in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean ± SE of duplicate determinations in 2 experiments).

of volatile compounds found by Carbonell et al. (2002) can be attributed to the higher number of cheeses studied, which were representative of 24 batches made during the 4 seasons of the year at 3 different dairies, with cheeses sampled throughout a longer ripening period. Cheeses studied in the present work contained high levels of adventitious microorganisms such as enterococci, micrococci, and gram-negative bacteria (Arqués



**Figure 4.** Carboxylic acids in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean  $\pm$  SE of duplicate determinations in 2 experiments).

et al., 2006). Without a detailed study of the microorganisms present in the cheeses studied by Carbonell et al. (2002), one can only hypothesize that the more diverse microbiota presumably present in 24 batches, compared with those in 2 batches, were mostly responsible for their higher number of volatile compounds.

High-pressure treatment of cheeses on d 2 had a significant effect on the relative abundances of 41 volatile compounds, most of which were found at lower levels in HP-treated cheeses than in control cheese. The main microbial groups were considerably reduced by HP treatment of these cheeses on d 2. Thus, log counts of lactic acid bacteria on d 3 were 8.56 for control cheese, 7.15 for cheese treated at 300 MPa, and 6.60 for cheese treated at 400 MPa, whereas the respective log counts for gram-negative bacteria were 8.45, 5.31, and 4.49 (Argués et al., 2006). The lower levels of viable bacteria in HP-treated cheeses most probably hindered the formation of volatile compounds by microorganisms. Also, significantly lower aminopeptidase activity values were found on d 3 in the HP-treated cheeses than in control cheese (Garde et al., 2007). This adverse effect of HP treatment on enzymes, previously reported by other authors (Casal and Gómez, 1999; Malone et al., 2003), might have affected the formation of volatile compounds in the cheese. Saldo et al. (2003) reported lower levels of FFA and volatile compounds in pasteurized

goat's milk Garrotxa cheese treated at 400 MPa for 5 min at 14°C on d 1 than in untreated cheese. In addition, Jin and Harper (2003) observed that some FFA decreased in Swiss cheese slurries treated on the day of manufacture at 345 MPa for 30 min or 550 MPa for 10 min in comparison with a control slurry. Juan et al. (2007) reported that acids, alcohols, ketones, aldehydes, and sulfur compounds were generally present at lower levels in pasteurized ewe's milk cheese treated at 300, 400, or 500 MPa for 10 min at 12°C on d 1 or 15 than in cheese treated at 200 MPa and in control cheese. According to Avila et al. (2006a), some volatile compounds (hexanal, 3-hydroxy-2-pentanone, 2-hydroxy-3pentanone, and hexane) were increased by HP treatment of 15-d-old Hispánico cheese at 400 MPa, whereas others (ethanal, ethanol, 1-propanol, ethyl acetate, ethyl butanoate, ethyl hexanoate, 2-pentanone, and butanoic acid) declined.

In the present work, HP treatments of 2-d-old La Serena cheese increased the levels of volatile compounds such as 3-methylbutanal, 2-propanol, 2-pentanol, 3-methyl-3-buten-1-ol, and 3-methyl-2-buten-1-ol during ripening. The higher levels of 3-methylbutanal in cheeses HP treated on d 2 than in control cheese can be explained by the higher Leu concentrations of the former cheeses (Garde et al., 2007), because 3-methylbutanal is formed from Leu by transamination or



**Figure 5.** Esters in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high-pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean ± SE of duplicate determinations in 2 experiments).

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**Figure 6.** Pentane, hexane, and octane in 3-, 30-, and 60-d-old La Serena cheeses, untreated or high pressure (HP) treated on d 2 at 300 or 400 MPa, or on d 50 at 300 or 400 MPa. Data represent relative abundances against cyclohexanone (mean  $\pm$  SE of duplicate determinations in 2 experiments).

Strecker degradation (Christensen et al., 1999). An increase in the level of branched-chain aldehydes in pasteurized ewe's milk cheese treated at 300 MPa for 10 min on d 1, which contained the highest amount of free AA, was reported by Juan et al. (2007). The alcohol 2-propanol is formed through the reduction of 2-propanone. Some strains of lactococci and lactobacilli have been reported to consume 2-propanone in milk cultures (Imhof et al., 1995). La Serena cheeses HP treated on d 2 exhibited lower microbial counts than

Table 1. Sensory characteristics of 60-d-old La Serena cheeses, high-pressure (HP) treated on d 2 or 50, at 300 or 400 MPa for 10 min

		HP treated on d 2		HP treated on d 50	
$Characteristic^1$	Control	300 MPa	400 MPa	300 MPa	400 MPa
Odor					
Quality	$5.97 \pm 0.21^{\rm a}$	$5.59 \pm 0.27^{\rm a}$	$5.55 \pm 0.37^{\rm a}$	$6.17 \pm 0.19^{\rm a}$	$5.77 \pm 0.27^{\rm a}$
Intensity	$6.23 \pm 0.23^{\rm a}$	$5.81 \pm 0.34^{\rm a}$	$6.18 \pm 0.29^{\rm a}$	$6.26 \pm 0.29^{\rm a}$	$5.98 \pm 0.29^{\rm a}$
Family "lactic"	$3.38 \pm 0.40^{\rm a}$	$2.90 \pm 0.58^{\rm a}$	$3.52 \pm 0.53^{\rm a}$	$3.53 \pm 0.55^{\rm a}$	$3.01 \pm 0.57^{\rm a}$
Family "vegetable"	$0.80 \pm 0.23^{\rm a}$	$0.70~\pm~0.36^{\rm a}$	$1.06 \pm 0.39^{\rm a}$	$1.11 \pm 0.46^{\rm a}$	$1.57 \pm 0.52^{\rm a}$
Family "floral"	$0.85 \pm 0.34^{\rm a}$	$0.61 \pm 0.41^{\rm a}$	$0.75 \pm 0.43^{\rm a}$	$0.77 \pm 0.42^{\rm a}$	$0.73 \pm 0.43^{\rm a}$
Family "animal"	$3.84 \pm 0.44^{\rm a}$	$4.33 \pm 0.53^{\rm a}$	$3.86 \pm 0.62^{\rm a}$	$4.06 \pm 0.62^{\rm a}$	$3.90 \pm 0.55^{\rm a}$
Aroma					
Quality	$6.27 \pm 0.20^{ m b}$	$5.86~\pm~0.20^{ m ab}$	$4.99 \pm 0.27^{\rm a}$	$6.22 \pm 0.21^{ m b}$	$5.71 \pm 0.26^{\rm ab}$
Intensity	$6.39 \pm 0.23^{ m bc}$	$5.56~\pm~0.39^{ m ab}$	$4.94 \pm 0.46^{\rm a}$	$6.69 \pm 0.34^{\circ}$	$6.18 \pm 0.37^{\rm bc}$
Family "lactic"	$3.09 \pm 0.37^{\rm a}$	$3.24 \pm 0.57^{\rm a}$	$2.70 \pm 0.51^{\rm a}$	$2.87 \pm 0.63^{\rm a}$	$2.75 \pm 0.54^{\rm a}$
Family "vegetable"	$1.06 \pm 0.31^{\rm a}$	$1.05 \pm 0.48^{\rm a}$	$1.21 \pm 0.45^{\rm a}$	$1.23 \pm 0.47^{\rm a}$	$1.35 \pm 0.53^{\rm a}$
Family "floral"	$0.86 \pm 0.35^{\rm a}$	$0.85 \pm 0.48^{\rm a}$	$0.54 \pm 0.34^{\rm a}$	$0.85~\pm~0.48^{\rm a}$	$0.77 \pm 0.45^{\rm a}$
Family "animal"	$4.58~\pm~0.42^{\rm a}$	$3.72~\pm~0.63^a$	$3.69~\pm~0.59^{\rm a}$	$4.87~\pm~0.51^{\rm a}$	$3.77 \pm 0.64^{\rm a}$

<sup>a-c</sup>Means in the same row with different superscripts differ (P < 0.05).

 $^{1}$ Mean ± SE (n = 24) of scores from 12 trained panelists in 2 cheese-making experiments on a 10-point scale.

#### HIGH-PRESSURE TREATMENT AND VOLATILES OF LA SERENA CHEESE

_	Principal component 1	Principal component 2
Item	(45.4% variance)	(16.9% variance)
Volatile compound		
Unsaturated alcohols	0.840	0.342
2-Butanol	0.794	0.524
Propyl esters	0.793	0.537
2-Butanone	0.725	0.634
Branched-chain esters	0.712	-0.023
2-Alcohols (except 2-butanol)	-0.695	-0.292
Branched-chain alcohols	0.685	0.039
Branched-chain aldehydes	-0.678	-0.259
n-Alcohols	0.671	0.636
2-Methyl ketones (except 2-butanone)	0.518	0.424
Aldehydes	0.280	0.555
Ethyl esters	0.039	0.505
Sensory characteristic		
Aroma intensity	0.940	0.068
Aroma quality	0.891	-0.403
Aroma family "animal"	0.710	-0.208
Odor quality	0.525	0.063
Aroma family "lactic"	0.232	-0.799
Aroma family "vegetable"	-0.194	0.721
Aroma family "floral"	0.188	0.692
Odor intensity	0.232	0.613

**Table 2.** Correlation coefficients of volatile compounds and sensory characteristics of La Serena cheeses with principal components in the principal components analysis with Varimax rotation



Figure 7. Principal components analysis plot showing the distribution of volatile compounds and sensory characteristics of 60-d-old cheeses.

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**Figure 8.** Distribution of 60-d-old La Serena cheeses in the plane defined by principal components 1 and 2 of the principal components analysis in Figure 7. Untreated cheese  $(\bigcirc)$ , high-pressure-treated cheeses on d 2 at 300  $(\triangle)$  or 400 MPa  $(\Box)$ , and high-pressure-treated cheeses on d 50 at 300  $(\blacktriangle)$  or 400 MPa  $(\blacksquare)$ .

control cheese (Arqués et al., 2006). This would leave a higher amount of 2-propanone available for its reduction to 2-propanol, which was found at higher levels in HP-treated cheeses.

Odor quality and intensity were unaffected by HP treatments on d 2, despite the significantly lower levels of volatile compounds such as 2-butanone, 2-pentanone, 3-hydroxy-2-butanone, 1-propanol, 1-butanol, 1-hexanol, 2-butanol, 2-propen-1-ol, ethyl butanoate, ethyl lactate, propyl esters, and 1-methylpropyl acetate on d 60 in cheeses HP treated on d 2. However, HP treatment of cheese on d 2 at 400 MPa had a negative effect on its aroma quality and intensity. A negative effect of HP treatment of 15-d-old Hispánico cheese at 400 MPa on odor quality and intensity, and on aroma intensity has been reported by Avila et al. (2006a). In the present work, differences in aroma for the same volatile profiles of cheeses apparently were more easily perceived by panelists than differences in odor. This, together with the fact that the more severe treatment had a more marked effect on the levels of some volatile compounds, would explain the significant differences in aroma quality and intensity of cheese treated on d 2 at 400 MPa, but not at 300 MPa, in comparison with control cheese.

High-pressure treatments on d 50 had a minor effect on volatile compounds of 60-d-old cheeses. Only butanoic acid and ethyl decanoate increased with treatment at 400 MPa on d 50, whereas 2,3-butanedione, 3-hydroxy-2-butanone, 3-methyl-3-buten-1-ol, and ethyl butanoate decreased with at least one of the HP treatments on d 50. Changes in the levels of volatile compounds brought about by HP treatments on d 50 were not sufficient to affect the sensory characteristics of 60d-old La Serena cheeses. Even though HP treatments on d 50 might have altered the viability of microorganisms and the activity of enzymes, the 10 d that elapsed until the determination of volatile compounds and the sensory analysis on d 60 was probably too short a period to exert a marked influence on the levels of volatile compounds or the scores of sensory characteristics.

In the PCA, unsaturated alcohols, 2-butanol, propyl esters, 2-butanone, branched-chain esters, branchedchain alcohols, n-alcohols, 2-methyl ketones, aroma intensity and quality, the aroma descriptor "animal," and odor quality were shown to correlate positively with principal component 1, whereas 2-alcohols and branched-chain aldehydes correlated negatively. "Animal" flavor notes have previously been correlated with 2-butanol and some esters in La Serena cheese (Carbonell et al., 2002) and have been associated with 2methyl ketones such as 2-heptanone (Curioni and Bosset, 2002). n-Alcohols, 2-butanone, aldehydes, propyl esters, 2-butanol, ethyl esters, the aroma descriptors "vegetable" and "floral," and odor intensity correlated positively with principal component 2, whereas the aroma descriptor "lactic" correlated negatively. "Fruity" flavor notes have previously been described for n-alcohols, aldehydes, and esters (Curioni and Bosset, 2002). The correlation between levels of esters and the intensity scores of aroma and odor is presumably favored by the low detection threshold of these compounds (Curioni and Bosset, 2002). The panelists considered aroma intensity and the aroma descriptor "animal" as quality enhancers.

# CONCLUSIONS

High-pressure treatment of 2-d-old raw milk La Serena cheese at 300 or 400 MPa decelerated the formation of some volatile compounds and had a negative effect on odor intensity, aroma quality, and aroma intensity. However, HP treatment on d 50 influenced only the relative abundance of 6 volatile compounds and did not affect sensory characteristics. Because both HP treatments on d 2 and 50 were effective in significantly reducing the counts of undesirable contaminating microorganisms in La Serena cheese, we concluded that HP treatment at 400 MPa on d 50 is a valuable tool for controlling undesirable microorganisms without altering the sensory characteristics of La Serena cheese.

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