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Volatile profile of mango (*Mangifera indica* L.), as affected by osmotic dehydration

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Abstract

The effect of osmotic dehydration on the volatile fraction of mango fruit was studied. Osmotic treatments were carried out at atmospheric pressure (OD) and by applying a vacuum pulse (PVOD). Sucrose at 35, 45, 55 and 65 °Brix was used as osmotic solution until reaching 20 or 30 °Brix in the liquid phase of dehydrated mango. Volatile compounds of fresh and dehydrated samples were obtained by simultaneous distillation–extraction, and analyzed by GC–MS. In general, osmotic dehydration provoked changes in the concentration of analyzed compounds to different extents, depending on process conditions. The use of highly concentrated osmotic solutions, and the high level of sample osmodehydration, induced losses of volatiles with respect to the fresh samples. On the other hand, more heavily diluted solutions and shorter treatment times (lower osmodehydration level) could give rise to the enhancement of volatile production. In these cases, sample mass loss was reduced during treatment since sugar gain was promoted against water loss.

Keywords: Mangoes; Osmotic dehydration; GC–MS; Volatile compounds; Minimally processed fruit

1. Introduction

Mango fruit is one of the most widely sold tropical fruits in Europe and its commercialization, as fresh cut fruit, is gaining importance in the market. However, fresh-cut fruits have a very short shelf life because of the difficulties in preserving their fresh-like quality (Soliva-Fortuny & Martın-Belloso, 2003). Minimally processed fruits are products that contain living tissues, which have suffered minor changes from their fresh state. Some studies have reported quality changes, which occurred in fresh cut products, such as physiological and physicochemical alterations (Watada, Ko, & Minott, 1996) and specifically in fresh cut mango (Beaulieu & Lea, 2003). Some basic operations of minimal processing, such as peeling and slicing, induce quality changes due to the lesions produced in the tissue (Brecht, 1995; Watada et al., 1996) which, among other things, enhance enzyme activity (Yu & Yang, 1980) and provoke physiological changes (Balwin, Nisperos-Carriedo, & Beaker, 1995), thus giving rise to a reduction in the shelf life of minimally processed fruits when compared to the whole pieces.

An alternative for reducing the above-mentioned undesired changes could be the partial dehydration of the cut fruit by applying mild osmotic dehydration treatments, which slightly reduce the fruit water activity, thereby limiting the deteriorative process, especially in the more external cells, which dehydrate to a greater degree and can act as a barrier for the rest of the tissue (Tovar, Garcıa, & Mata, 2001b).

Osmotic dehydration (OD) involves the immersion of fruit in concentrated sugar solutions, where both partial dehydration of the tissue and solid uptake take place. Mass transfer rates during OD depend on factors such as temperature, concentration of osmotic medium, size and geometry of the samples and degree of agitation of the solution. In the osmotic processes, application of vacuum for a short period at the beginning of the process (vacuum pulse

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osmotic dehydration: PVOD) had beneficial effects on process kinetics and fruit quality in many fruits (Fito, 1994; Fito & Chiralt, 2000), affecting physical and transport properties of the plant tissue. The increase in process rate in PVOD allows us to work at a mild temperature, thus preserving product quality attributes. Osmotic solution (OS) concentration and viscosity greatly affect the product response to vacuum impregnation (VI) (Barat, Fito, & Chiralt, 2001; Cháfer, González-Martınez, Ortolá, Chiralt, & Fito, 2001) and the osmodehydration kinetics, as well as the internal ratio, water loss: sugar gain, in the product, that in turn has a great influence on product characteristics. The major sugar in mango is sucrose (Gil et al., 2000), and so, it is expected that the osmotic treatments using solutions of this sugar are those that alter the product sensory properties the least. The kinetics of mango osmodehydration using sucrose solutions, as a function of solution concentration and application of vacuum pulse have been studied by Giraldo, Talens, Fito, and Chiralt (2003). The effect of osmotic treatments on several properties of the tissue (enzymatic activity, respiration rate, ethylene production and organic acid evolution) has been reported by Tovar, Garcıa, and Mata (2001a, 2001b). Nevertheless, no previous studies were found on the effect of osmotic treatments on mango volatile profile, as related to the fruit aroma.

The fruit flavour is an important quality factor that influences consumer acceptability and, for this reason, its study is relevant in the minimally processed food product. The volatile compounds that are involved in the fruit flavour are produced through metabolic pathways during ripening, harvest, post-harvest and storage, and depend on many factors related to the species, variety and type of technological treatments (Ibáñez, López-Sebastián, Ramos, Tabera, & Reglero, 1998). Mango aroma is mainly formed by a complex mixture of compounds, but some authors consider terpenes, especially 3-carene, as the most important aroma constituents, due to the high percentage in the volatile fraction (50–60%) (Andrade, Maia, & Zoghbi, 2000). The terpene hydrocarbons are considered to be important contributors to the flavour of Florida mango varieties, such as "Keitt", "Kent" and "Tommy Atkins" (Malundo, Baldwin, Ware, & Shewfelt, 1997). Engel and Tressl (1983) identified the monoterpenes as an important class of volatiles contributing to the flavour of Florida, Brazilian and Venezuelan mango varieties, in contrast to Indian varieties, which have more oxygenated volatile compounds, such as esters, furanones, and lactones. The use of suitable technologies, such as osmotic dehydration at mild temperatures, permits the production of minimally processed food, to a great extent preserving the flavour and colour of fresh fruit (Heng, Guilbert, & Cuq, 1990).

In this paper, the influence of osmotic dehydration on the volatile profile of mango, concentrated up to 20 and 30 °Brix, was analyzed by considering the osmotic solution concentration and the application, or not, of a vacuum pulse at the beginning of the process to promote sample degasifying and impregnation as process variables.

2. Materials and methods

2.1. Sample preparation

Mango (*Mangifera indica* L.) *Kent var*. fruits, selected on the basis of a similar ripening degree, were purchased in a local market. Two slices, parallel to the stone, were cut from each fruit and cylinders (1.5 cm height and 2.0 cm diameter) were taken with a core borer from these. Treatments were carried out on cylinders (10 for each treatment), coming from 10 different mangoes that were used for half the treatments. So, two batches, of 10 mangoes in each, were used for all the experiments. All samples were characterized as to moisture, soluble solids, water activity and volatile fraction.

2.2. Osmotic treatments

Mango samples were submitted to osmotic dehydration treatments, at 30 °C, using four sucrose concentrations: 35, 45, 55 and 65 °Brix, prepared by adding sucrose (commercial sugar) to distilled water. Equipment with pressure and temperature control, and osmotic solution (OS) recirculation (80 rpm) were used. Osmotic dehydration was carried out at normal pressure (OD) and by applying a vacuum pulse (PVOD) (10 min, 50 mbar) at the beginning of the process (Fito & Chiralt, 1995). The samples were immersed in the osmotic solution for long enough to reach 20 or 30 °Brix in the fruit liquid phase. Therefore, 16 different treatments were considered: OD 35-20, OD 35-30, OD 45-20, OD 45-30, OD 55-20, OD 55-30, OD 65-20; OD 65-30, PVOD 35-20, PVOD 35-30, PVOD 45-20, PVOD 45-30, PVOD 55-20, PVOD 55-30, PVOD 65-20 and PVOD 65-30. Mango batch 1 was used for treatments with 45 and 65 °Brix osmotic solutions and batch 2 for treatments with 35 and 55 °Brix osmotic solution. Process times, shown in Table 1, were determined according to a previous kinetic study carried out under the same conditions (Giraldo et al., 2003). Due to the large number of samples to be analyzed according to the experimental design, all samples were frozen and stored at −40 °C prior to analysis in sealed polyethylene plastic bags for no more than 2 days in order to provoke the minimum alterations in volatiles.

2.3. Analytical determinations

Processed samples were characterized as to mass and water loss and sugar gain during osmotic treatment. Likewise, fresh samples from each batch and all processed samples were analyzed, as well as to their volatile fraction, as described below.

Moisture content was determined by drying to constant weight at 60 °C (method 20.013 AOAC, 1980). Soluble sol-

Table 1
Process time and composition changes of the samples in the different treatments [mass fraction of water (x_w), soluble solids (x_s), and liquid phase soluble solid content (z_s), weight loss (ΔM) and water loss (ΔM_w) and sugar gain (ΔM_s)]

Treatment	Process time (min)	x_w	x_s	z_s	ΔM[a]	ΔM_w[a] (kg/kg)	ΔM_s[a] (kg/kg)
FRESH 1[b]		0.825 ± 0.002	0.156 ± 0.004	0.160 ± 0.004			
FRESH 2[c]		0.823 ± 0.005	0.157 ± 0.001	0.153 ± 0.001			
OD 35-20	129	0.783 ± 0.001	0.200 ± 0.001	0.204 ± 0.001	−0.042 ± 0.001	−0.076 ± 0.001	0.035 ± 0.001
OD 45-20	29	0.774 ± 0.007	0.209 ± 0.005	0.204 ± 0.004	−0.104 ± 0.004	−0.129 ± 0.003	0.030 ± 0.005
OD 55-20	17	0.780 ± 0.002	0.204 ± 0.005	0.207 ± 0.004	−0.106 ± 0.001	−0.128 ± 0.001	0.025 ± 0.005
OD 65-20	1	0.766 ± 0.003	0.214 ± 0.002	0.208 ± 0.002	−0.151 ± 0.002	−0.172 ± 0.001	0.024 ± 0.002
PVOD 35-20	39	0.775 ± 0.008	0.204 ± 0.004	0.209 ± 0.005	0.073 ± 0.019	0.007 ± 0.023	0.062 ± 0.001
PVOD 45-20	3	0.787 ± 0.002	0.195 ± 0.001	0.190 ± 0.001	0.100 ± 0.001	0.042 ± 0.002	0.056 ± 0.002
PVOD 55-20	2	0.781 ± 0.004	0.201 ± 0.003	0.205 ± 0.004	−0.102 ± 0.001	−0.124 ± 0.002	0.024 ± 0.003
PVOD 65-20	2	0.792 ± 0.003	0.193 ± 0.001	0.189 ± 0.001	−0.099 ± 0.022	−0.110 ± 0.015	0.018 ± 0.005
OD 35-30	901	0.696 ± 0.007	0.293 ± 0.009	0.296 ± 0.008	−0.155 ± 0.001	−0.237 ± 0.006	0.091 ± 0.008
OD 45-30	347	0.681 ± 0.001	0.306 ± 0.001	0.300 ± 0.002	−0.253 ± 0.002	−0.314 ± 0.001	0.072 ± 0.001
OD 55-30	147	0.682 ± 0.003	0.305 ± 0.005	0.309 ± 0.004	−0.296 ± 0.001	−0.345 ± 0.001	0.058 ± 0.004
OD 65-30	224	0.679 ± 0.009	0.309 ± 0.009	0.303 ± 0.009	−0.344 ± 0.003	−0.377 ± 0.009	0.046 ± 0.005
PVOD 35-30	823	0.701 ± 0.002	0.284 ± 0.002	0.289 ± 0.002	−0.125 ± 0.002	−0.212 ± 0.004	0.092 ± 0.001
PVOD 45-30	300	0.703 ± 0.002	0.290 ± 0.001	0.286 ± 0.001	−0.175 ± 0.003	−0.243 ± 0.001	0.084 ± 0.001
PVOD 55-30	181	0.682 ± 0.005	0.300 ± 0.005	0.306 ± 0.005	−0.274 ± 0.002	−0.330 ± 0.003	0.061 ± 0.004
PVOD 65-30	148	0.671 ± 0.003	0.314 ± 0.001	0.307 ± 0.001	−0.335 ± 0.001	−0.377 ± 0.001	0.052 ± 0.001

OD: osmotic dehydration at atmospheric pressure; PVOD: pulsed vacuum osmotic dehydration; 65, 55, 45 and 35 correspond to °Brix of the osmotic solution; 20 and 30 correspond to °Brix of the sample liquid phase.
[a] (kg/kg of initial sample).
[b] Used for treatments with 35 and 55 °Brix osmotic solutions.
[c] Used for treatments with 45 and 65 °Brix osmotic solutions.

ids were measured in samples, previously homogenized, using a refractometer (ATAGO model NAR-3T) at 20 °C.

2.4. Isolation and analysis of volatile compounds

Volatile components of fresh and processed mango samples were isolated by using a combined simultaneous distillation–extraction (SDE) technique with pentane as solvent (Godefroot, Sandra, & Verzele, 1981), in a J&W Simultaneous Steam Distillation–Extraction Apparatus obtained from Fisher Scientific UK™ Ltd (Loughborough, Leics. England), by using the methodology described in a previous paper (Escriche, Chiralt, Moreno, & Serra, 2000; Talens, Escriche, Martinez-Navarrete, & Chiralt, 2003). Despite the fact that some authors reported artifacts when boiling took place for several hours (Werkhoff, Güntert, Krammer, Sommer, & Kaulen, 1998), this problem has probably been minimized by shortening the boiling time and using smaller amounts of sample. In fact, this technique (SDE) has been used in recent works to analyse volatile compounds in mango (Andrade et al., 2000), strawberry (Zabetakis, Koulentianos, Orruño, & Boyes, 2000) and papaya (Almora et al., 2004). In each analysis, 70 ml of bi-distilled water, 35 g of sample, previously homogenized using an Ultra-Turrax macerator T 25 model, and 100 μl of camphor (internal standard) at 200 μg/l for fresh samples and 75 μl of the same standard for processed samples, were put into a 500 ml round-bottomed flask. The flask was held in an ultrasonic bath for 2 min to totally disintegrate the sample and was then introduced into the extraction equipment oil bath and heated until boiling at around 100 °C. The 50 ml heart flask containing 3 ml of pentane was put into a water bath at 40 °C. The steam of both flasks was condensed in the common refrigerated "U-tube" of the equipment. After 30 min of distillation, the content of the U-tube was collected in an airtight sealed tube and frozen at −18 °C to facilitate the separation of the organic fraction (which is liquid and has lower density at −18 °C) where aromatic compounds were dissolved. This organic phase was concentrated, under nitrogen stream, up to a final volume of approximately 50 μl. The analysis was conducted on a GC–MS Finnigan TRACE™ MS (TermoQuest, Austin, USA) chromatograph (Norwalk, CT, USA) with a fused silica capillary column (DB-5, 30 m; 0.32 mm i.d.; J&W Scientific, Cromlab, Spain). The oven temperature was programmed from 40 °C to 60 °C at a ramp rate of 2 °C/min; afterwards this was increased to 260 °C, at a ramp rate of 4 °C/min, and the final holding time was 2 min (Andrade et al., 2000). Helium was used as a carrier gas at a constant flow rate of 2 ml/min. A 5 μl extract was injected with a split ratio of 1:16. The MS fragmentation was performed by electronic impact EI^+ at 70 eV, and scan mode was between 35 and 450 mass units; the scan rate was 2.5 scans/s.

Compounds were identified by comparing their mass spectra with those of the NIST library. Another confirmation was obtained by determining their relative retention indices, using *n*-alkanes, and comparing them to those reported in the literature (Kondjoyan & Berdagué, 1996). The identity of some selected compounds was further verified by comparing their mass spectra and retention time with those obtained for authentic standard compounds. In order to correct any losses that could occur during

analytical process, quantification of compounds was carried out using the internal standard method (camphor). Precision of the SDE extraction (and subsequent analysis) was determined by the average variation coefficient of the within-day and between-day variations of samples. The accuracy of the method was assessed by adding a known concentration of all the previously identified standards to the sample during the initial preparation. The overall recovery of all standards varied between 90% and 92% for all compounds.

A minimum of two extracts was obtained for each sample, both fresh and processed mangoes, and each extract was analyzed in triplicate. All reagents were of analytical grade. Standard compounds for defining GC Kovat's indices and mass spectra were purchased from Sigma–Aldrich S.A. (Madrid, Spain).

2.5. Statistical analysis

The data were analysed by use of multivariate techniques, applying the software Unscrambler version 9.2 (CAMO Process AS, Oslo, Norway). The variables were weighted with the inverse of the standard deviation of all objects in order to compensate for the different scales of the variables (Martens & Martens, 2001).

Partial least square regression, PLSR2, (Martens & Næs, 1989) was applied to describe the relationships among the volatile compounds and compositional variables. In addition, this procedure is useful for each treatment volatile profile determination and the evaluation of resemblances and differences among processes. In this analysis, mass loss and water content loss values have been taken as absolute values in order to simplify the comprehension of the model prediction.

3. Results and discussion

3.1. Physicochemical changes induced by osmotic treatments

Table 1 shows the mass fraction of water (x_w) and soluble solids (x_s) of processed mango samples reached in each treatment. Fresh samples showed $x_w = 0.824 \pm 0.003$ and $x_s = 0.156 \pm 0.003$. The mass fraction of soluble solids in the fruit liquid phase (water plus solutes) reached in the different treatments was 0.200 ± 0.003 and 0.300 ± 0.004, respectively for each sample group, according to the experimental design. In Table 1, it can be observed that this concentration is reached with different water loss (ΔM_w) and sugar gain (ΔM_s) levels (defined according to Fito & Chiralt (1997), per kilograms of fresh fruit), depending on the process conditions. As expected, mass loss of the samples (as a result of the balance of water loss and solute gain) increased as the fruit concentration level increased but, for each level, the values ranged widely, depending on the osmotic solution concentration and the application, or not, of vacuum pulse. In both, OD and PVOD processes, mass loss increased when OS concentration increased and the vacuum pulse implied a reduction of mass loss; the more diluted the OS, the greater was the reduction and, even in samples treated with 35 and 45 °Brix OS, concentrated to 20 °Brix, a positive mass gain was observed.

This can be explained by the coupled action of different mass transport mechanisms (osmo-diffusional and hydrodynamic), which occurs to a different extent, in each case. Low viscosity (less concentrated), osmotic solutions and vacuum pulse favour the hydrodynamic gain of the osmotic solution in the tissue pores, which allows us to obtain a determined overall concentration in the sample with smaller water (and weight) loss (Fito, Chiralt, Barat, & Martınez-Monzó, 2002; Lazarides, Fito, Chiralt, Gekas, & Lenart, 1999). The sample impregnation with the osmotic solution, promoted by vacuum pulse, is also favoured when solution viscosity is low, in comparison with sample volume compression (coupled to impregnation) that prevails when the solution has high viscosity (Chiralt et al., 1999). Likewise, low osmotic solution concentration implies a smaller process driving force and the subsequent longer treatment times when sample capillary impregnation is promoted.

Concentration profiles developed in sample tissue will also be dependent on process conditions. The lowest OS concentration induces a flatter water distribution profile in the tissue, at a determined overall concentration of the sample, than highly concentrated solutions. Although there are a greater number of cell layers affected by the osmotic treatment, the changes provoked in them are less intense than when highly concentrated osmotic solutions are used. In affected cells, membranes can be denatured and solute diffusion allowed through a wider zone in the sample, thus promoting the net solute gain (Talens et al., 2003). Concentration profiles in PVOD samples will be flatter than in the corresponding OD ones, since the disturbance front in the tissue is in a more internal location due to the promoted action of hydrodynamic mechanisms and sample impregnation (Fito et al., 2002).

3.2. Changes in volatile profile

Fifty volatile compounds were identified in all samples. Due to the fact that samples were frozen stored before volatile fraction analysis, a previous evaluation of possible losses of volatile compounds due to freezing was carried out by comparing relative areas (to internal standard) of fresh and fresh-frozen samples (Table 2). Scant effect of frozen storage conditions could be observed, since the losses of volatiles were in the range of 7%.

To analyze the changes induced by treatments in volatile profile, just 10 of the most representative compounds found in Florida mango varieties (Andrade et al., 2000; Engel & Tressl, 1983; Malundo et al., 1997) were quantified in all samples: heptanal, benzaldehyde, nonanal, α-pinene, camphene, β-myrcene, α-phelandrene, 3-carene, limonene, α-linalool. Table 3 shows concentration values (μg/g) for fresh samples in the two batches used in this study. Variability of fresh fruit, in terms of quantified com-

Table 2
Identified compounds in fresh mango before and after freezing at −40 °C for 48h, and relative area to internal standard in both cases

Compound	Kovat's indices	Relative area		Compound	Kovat's indices	Relative area	
		Fresh	Frozen			Fresh	Frozen
Heptanal	886.1	0.271	0.247	Cinnamic acid-3-phenyl-ethyl ester	1418.3	0.217	0.200
α-Pinene	916.5	0.140	0.132	Germacrene D	1422.9	0.176	0.164
Camphene	928.4	0.046	0.042	Caryophyllene	1426.6	0.076	0.067
Benzaldehyde	937.5	0.088	0.080	α-Lanone	1428.9	0.113	0.105
Myrcene	973.8	0.226	0.212	Ledene	1438.2	0.044	0.040
α-Phellandrene	983.3	0.170	0.160	Phenol-2,4-di-*tert*-butyl	1459.8	0.227	0.214
3-Carene	988.9	6.814	6.752	Lauric anhydride	1512.7	0.086	0.079
α-Terpinen	980.6	0.041	0.037	Benzoic acid 4-(dimethylamino) ethyl ester	1638.3	0.533	0.521
Limonene	1008.4	0.260	0.249	Tetradecanal (myristaldehyde)	1650.0	0.857	0.823
τ-Terpinene	1022.5	0.066	0.060	Tetradecanoic acid (myristic acid)	1703.6	0.020	0.016
Octanal	1038.0	0.103	0.095	Tetranoic acid ethyl ester	1735.1	0.226	0.214
Cyclohexene	1052.0	0.480	0.470	Isopropyl myristate	1768.5	0.134	0.121
α-Linalool	1090.5	0.069	0.065	Hexadecanoic acid methyl ester	–	3.00	2.92
Nonanal	1090.5	0.540	0.495	9-Hexadecanoic acid	–	1.69	1.59
2,6-Nonadienal	1113.4	0.129	0.120	*n*-Hexadecanoic acid	–	0.039	0.036
2-Nonenal	1119.8	0.335	0.326	Hexadecanoic acid ethyl ester	–	0.780	0.720
Decanal	1160.9	0.229	0.219	Isopropyl palmitate	–	0.208	0.196
Benzothiazole	1166.8	0.155	0.152	Oleic acid methyl ester	–	3.386	3.312
Phenol-*m*-*tert*-butyl	1221.0	0.091	0.086	Hexadecadienoic acid methyl ester	–	0.374	0.357
2-Methoxy-4-vinylphenol	1228.9	0.020	0.016	Nonanoic acid-9-(*o*-propylphenyl) methyl ester	–	0.202	0.191
Docosane	1238.9	0.023	0.018	Stearic acid	–	0.253	0.244
Copaene	1260.1	0.099	0.090	Octadecanoic acid ethyl ester	–	0.270	0.260
α-Cubene	1260.4	0.029	0.025	Acetic acid octadietyl ester	–	0.063	0.051
α -Damascenone	1264.9	0.636	0.592	Squalene	–	0.220	0.207
α-Caryophyllene	1281.4	1.656	1.57				

Table 3
Volatile compounds quantified (μg/g raw sample) in fresh mango batches

Compounds	Concentration μg/g raw sample			α
	Batch 1	Batch 2	Mean	
Aldehydes				
Heptanal	0.019 ± 0.000	0.034 ± 0.001	0.027 ± 0.009	*
Benzaldehyde	0.008 ± 0.001	0.007 ± 0.001	0.008 ± 0.001	n.s.
Nonanal	0.053 ± 0.002	0.035 ± 0.002	0.044 ± 0.010	*
Terpenes				
α-Pinene	0.011 ± 0.001	0.009 ± 0.000	0.010 ± 0.001	n.s.
Camphene	0.002 ± 0.000	0.003 ± 0.000	0.002 ± 0.001	*
β-Myrcene	0.013 ± 0.001	0.020 ± 0.003	0.017 ± 0.004	n.s.
α-Phelandrene	0.006 ± 0.001	0.010 ± 0.002	0.008 ± 0.002	n.s.
3-Carene	0.300 ± 0.060	0.500 ± 0.050	0.401 ± 0.130	n.s.
Limonene	0.011 ± 0.002	0.016 ± 0.003	0.013 ± 0.004	n.s.
Monoterpenic alcohol				
α-Linalool	0.009 ± 0.001	0.005 ± 0.001	0.007 ± 0.003	*

n.s., not significantly different.
* $\alpha < 0.05$.

pounds, was very low, as reflected by the ANOVA carried out for each volatile component by considering the batch factor. Although four (heptanal, nonanal, camphene and α-linalool) of the 10 compounds showed statistically significant differences (at 95% confidence level), these were reasonably small. As also stated by other studies on mango volatiles (Andrade et al., 2000; Idsteim & Schreier, 1985; MacLeod & Gonzalez, 1982; MacLeod & Pieris, 1984), the highest concentration among the identified compounds was shown by 3-carene (0.40 ± 0.13 μg/g fresh fruit); this concentration represented 74.7% of the total quantified volatiles. The others fluctuated between 0.002 and 0.044 μg/g fresh fruit, obtained for camphene and nonanal, respectively (see Table 3).

The effect of osmotic treatment on the volatile profile of mango was evaluated through the concentration changes induced by the treatment in each compound. In this sense, for each compound, the mean value of fresh sample concentration (C_o) was subtracted from the value of processed samples (C) ($[C - C_o]$, for each replicate), both C and C_o being expressed as μg/g fresh sample.

Fig. 1 shows the concentration changes (μg/g fresh sample) induced by osmotic treatments for all quantified components. Osmotic treatments promoted changes in the volatile profile of mango, as are reported in previous works for strawberry (Escriche et al., 2000) and kiwi (Talens et al., 2003).

In general, when samples were osmodehydrated to 30 °Brix, a decrease in the volatile concentration was observed, especially in PVOD processed samples, 3-carene and nonanal being the most affected compounds. Nevertheless, in 20 °Brix samples, an increase in some volatiles was observed, due to the osmotic treatment. In these cases, treatments carried out using 45 °Brix solution involved the lowest changes in volatile profile. When 65 °Brix sucrose was used, the greatest losses of volatile compounds occurred in all cases. Treatments PVOD 35-20 and OD 55-20 gave the greatest increases in volatiles (in 9 of the 10 quantified compounds, there is an increase in concentration).

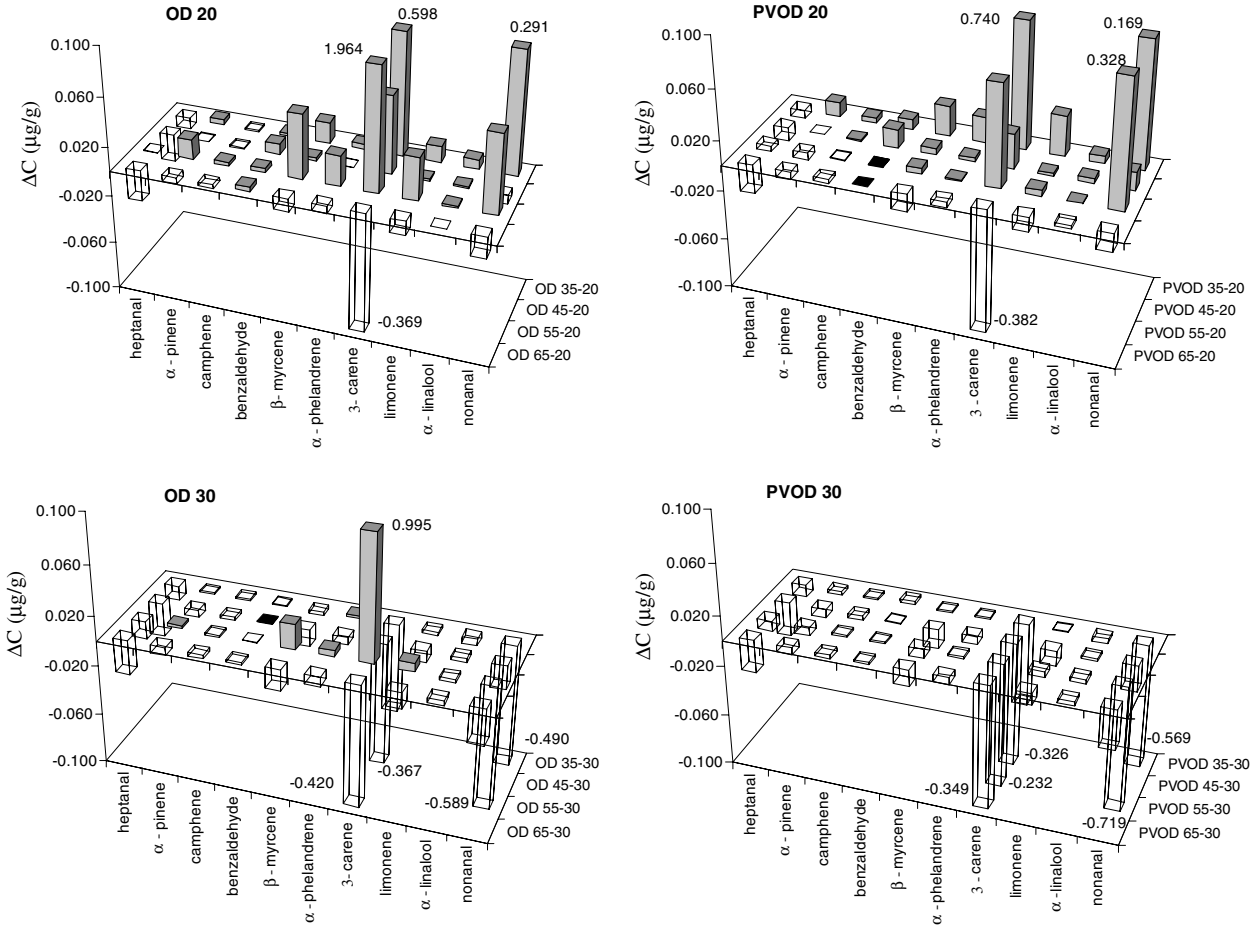

Fig. 1. Change of concentration ($\Delta C = C - C_o$) in the major mango fruit volatile compounds (μg/g raw sample) due to the osmotic treatments. (C_o) mean value of fresh sample concentration. (C) mean value of processed samples concentration. White bars represent negative values and dark bars positive values. Concentration for 3-carene and nonanal were specified to adapt the y-scale to the rest of the values.

Fig. 2 shows the effect of osmotic solution concentration, vacuum pulse and sample dehydration level on the changes in concentration of different quantified compounds. As far as the effect of treatments on the different compound families is concerned, a similar pattern was observed for the analyzed terpenic compounds of mango (α-pinene, camphene, β-myrcene, α-phelandrene, 3-carene and limonene). The maximum levels of terpenes are generated in samples of 20 °Brix treated at atmospheric pressure with 55 °Brix sucrose. Nevertheless, no general pattern was observed for the different identified aldehydes. The behaviour of benzaldehyde, which is considered a key compound in the fruit fragrance (Ibáñez et al., 1998), shows that treatments carried out using 45 °Brix enhance its formation when samples reached 20 °Brix.

After the individual behaviour of each compound was studied, a multivariate analysis (PLS2 regression) was done, with the purpose of describing the volatile fraction behaviour as related to both, the compositional variables and the applied treatments.

Fig. 3 shows the PCA biplot obtained considering the complete series of determined compounds in the volatile profile (in both fresh and treated samples) and the different treatments. It was found that two principal components (PCs) explained 81% of the variations in the data set. The PC1 explains 67% of the variability, and PC2 explains the 14%. In this plot, the nearness between treatments indicates a similar behaviour of the sample aromatic profile after the process and the proximity among compounds means the degree of correlation between their changes in concentration during treatments (similar change pattern). All compounds are located on the right semi-plan in the plot. The location of treatments on the plot generates two groups, clearly differentiated: those that are located on the right side, which correspond to the conditions that lead to the highest volatile concentration, and those that are located on the left side which lead to lowest volatile concentrations. In this last group, a great proximity of treatments can be observed where samples were concentrated to 30 °Brix (except OD 55-30) and those where 65 °Brix osmotic solutions were used. This suggests a very similar impact of these treatments, that leads to a loss of volatile compounds, as related to fresh samples. The first group includes all treatments where 20 °Brix was reached in the samples, except those carried out with 65 °Brix solution. Treatments PVOD 45-20 and PVOD 55-20 are very

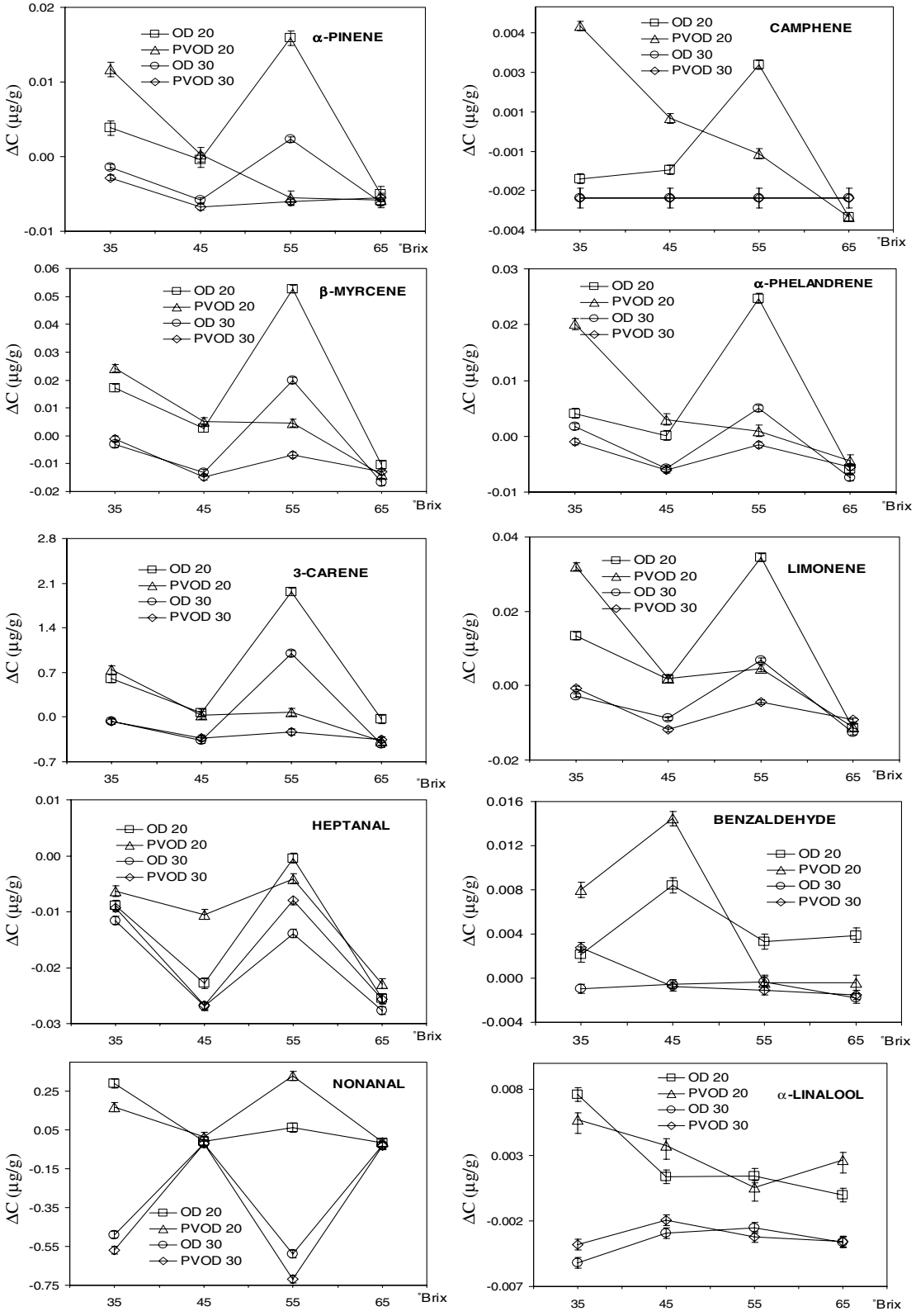

Fig. 2. Mean values and LSD intervals (95%) for difference of concentration of quantified compounds between osmodehydrated and fresh samples.

near to the fresh sample in the plot, in agreement with the fact that very similar volatile profiles was obtained in these cases. On the other hand, treatments OD 55-20 and PVOD 35-20 were those that showed a major volatile production.

According to the compound distribution along the second axis, a great separation between the major terpenic compounds (except camphene) and heptanal can be observed, thus indicating the different behaviour of both

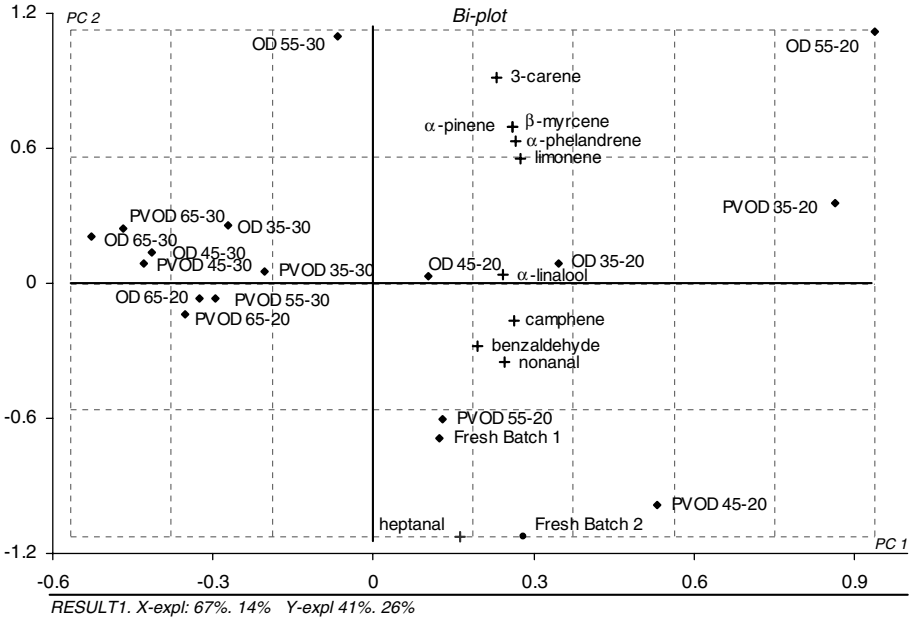

Fig. 3. Biplot for the treatments and volatile compounds (PC1 and PC2) obtained by means of the PLS2 analysis. Processes are identified by a black rhombus and compounds by a black cross.

compound groups. Heptanal, as a representative short chain aldehyde, characteristic of fresh fruits (Pfannhauser, 1988; Talens et al., 2003), is very close to fresh samples and samples treated in PVOD 45-20 and PVOD 55-20, which correspond to the process conditions that cause less alteration to the volatile profile of the samples. The proximity on the plot of terpenic compounds and treatments OD 55-20 and PVOD 35-20, indicates that these treatments facilitated the generation of these volatile compounds.

Fig. 4 shows the correlation loading plot for volatile concentration variables and those related to compositional changes in the samples (mass fraction of water (x_w) and soluble solids (x_s), liquid phase soluble solid content (z_s), weight loss (ΔM) and water loss (ΔM_w) and sugar gain (ΔM_s)). All compositional variables, except ΔM_s, showed significant correlations among the volatile compounds, as they are located between the two drawn ellipses. An increase of x_s, z_s, ΔM and ΔM_w variables is related to a decrease in all volatile compounds. However, an increase of x_w is associated with a development of all compounds, especially heptanal. This means that an increase in the concentration of sugar in samples implied greater losses of volatiles, especially heptanal.

The individual effect of process conditions (vacuum pulse, osmotic solution concentration and final sample concentration level) on final volatile profile is not clear, but there are interactions among them. Soluble solid concentrations of the samples seem to affect the volatile profile

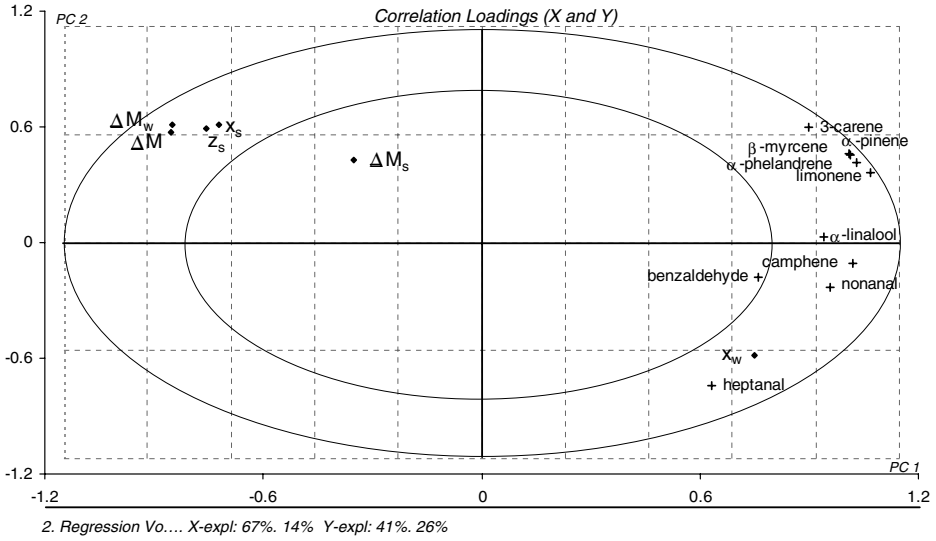

Fig. 4. Correlation loading plot (X and Y) for sample compositional variables and volatile compounds obtained by means of the PLS2 analysis.

more clearly. In this sense, a reduction of volatile concentration is induced when samples are more heavily osmodehydrated, Nevertheless, treatments with the most concentrated solution (65 °Brix) also lead to a great volatile concentration reduction, regardless of final Brix level reached in the samples.

The described behaviours could be the result of different phenomena responsible for changes in volatiles. These can occur due to lixiviation (diffusion from samples to the osmotic solution) and degradation–formation reactions, which take place in the tissue associated with cellular stress induced by the treatments. As far as lixiviation phenomena are concerned, treatment time has a great impact since longer treatments imply higher diffusion levels. Nevertheless, the degree of cellular alteration is also responsible for the physiological changes, depending on the process conditions. In this sense, different profiles and cellular alteration may be expected as a function of process variables (Albors, Salvatori, Andrés, Chiralt, & Fito, 1998; Salvatori, Albors, Andrés, Chiralt, & Fito, 1998). Osmotic stress, among other stress factors, promotes generation of volatile compounds in plant tissue, responsible for the fruit aroma by enzymatic action (Zabetakis & Holden, 1997). Previous studies have proved the role of enzymes in volatile development of strawberry when submitted to the osmotic process (Escriche et al., 2000).

Treatments carried out with highly concentrated solutions give rise to a clear solid concentration profile in the samples, which is associated with the degree of cellular alteration. On the other hand, diluted osmotic solutions induce flatter concentration profiles, where more cells are affected/altered, although less intensely. The application of vacuum pulse implies modifications in sample concentration profile since the osmotic solution penetrates deeply into the tissue, at the same time as the gases in the intercellular spaces are eliminated to a great extent, which may also affect biochemical pathways in stressed cells. On the other hand, the greater the overall sample concentration, the higher is the number of cells involved in the tissue changes. The number of altered cells and the degree of alteration will have a direct impact on the biochemical changes recorded through volatile analysis.

4. Conclusions

The volatile profile of mango was clearly affected by process conditions in osmotic treatments. In general, the use of highly concentrated osmotic solutions and the high level of sample osmodehydration induced losses of volatiles with respect to the fresh samples. On the other hand, more diluted solutions and shorter treatment times (lower osmodehydration level) can give rise to the enhancement of volatile production, which could be positive for the fruit aroma perception. Furthermore, in these cases, sample mass loss is reduced during treatment since sugar gain is promoted against water loss.

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