

# Free Volatile Components of Passion Fruit Puree Obtained by Flash Vacuum-Expansion

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Purple passion fruits were processed by the flash vacuum-expansion process. Volatile components were analyzed in purees from steam-heated fruits, steam-heated then vacuum-expanded fruits and their aromatic liquors, and fruit rind, in comparison with a reference single-strength juice. After steam heating, the puree was enriched in esters arising from the rind. Steam-heated then vacuum-expanded fruits yielded a puree impoverished in volatiles due to evaporation of  $\approx 10\%$  of water. These volatile compounds were mostly recovered in aromatic liquors.

**Keywords:** Purple passion fruit puree; flash vacuum-expansion process; volatile components; passion fruit rind

## INTRODUCTION

Flash vacuum-expansion is a process in which plant materials are first steam-heated to 60–90 °C, then instantly introduced in a vacuum chamber ( $\approx 2\text{--}5$  kPa) where they expand and disaggregate because of formation of microchannels inside the tissues and instantaneous evaporation of some constitution water and volatile aroma components, which are recovered with a condenser as aromatic liquors (Cogat, 1993, 1995). Steam heating induces a thermal denaturation of endogenous oxidases, whereas the whole process, performed with the absence of oxygen, prevents oxidation and subsequent browning of the products.

Passion fruit (*Passiflora edulis*; Passifloraceae), a highly favored fruit because of its unique exotic flavor, is composed of a thin hard exocarp, a thin purple mesocarp layer, and a white spongy endocarp surrounding a pulpy juicy edible aril containing seeds (Brat et al., unpublished results). It is traditionally processed by squeezing the inner juicy aril yielding strongly flavored orange-yellow juice. To obtain new intermediate food products, we processed entire purple passion fruits by flash vacuum-expansion (Brat et al., unpublished results). After refining the vacuum-expanded fruits with a rotative pulper, a purplish puree of strong consistency was obtained, which was explained by its high content in cell wall material arising from partial disaggregation of the rind with subsequent liberation of anthocyanins from the mesocarp layer.

Several comprehensive studies of the volatile flavoring constituents of fresh purple passion fruit juice have been undertaken (Parliment, 1972; Casimir et al., 1981; Werkhoff et al., 1998). However, because the vacuum-expanded puree includes part of the rind, in addition to the juice itself, it was desirable to investigate the

aroma components of this new product. Moreover, because the flash vacuum-expansion process includes two consecutive steps, steam heating then vacuum-expansion, we characterized volatiles of purees separately from steam-heated and vacuum-expanded fruits, aromatic liquors generated by the vacuum-expansion, and a rind puree in comparison with a single-strength juice. Because Winterhalter et al. (1987) had already shown that the composition of a flavor extract depended on the extraction procedure, we tested three techniques for isolation of volatiles: liquid–liquid extraction, simultaneous distillation–extraction (Likens and Nickerson, 1964), and vacuum headspace method (Werkhoff et al., 1998).

## MATERIALS AND METHODS

**Fruits.** Sound mature purple passion fruits (*P. edulis*) ( $31.8 \pm 10.2$  g;  $n = 40$ ) were air-freighted from Kenya, and after a thorough mixing of the fruits, four batches (10 kg) were constituted.

**Flash Vacuum-Expansion Equipment.** The equipment consisted of a cylindrical stainless steel steam-heating chamber ( $r = 6$  cm;  $h = 24$  cm;  $v = 2.7$  L) fed at normal pressure with a water-steam generator and coupled through a manual pneumatic valve (1 MPa; opening time, 0.5 s) to a cylindrical quartz vacuum vessel ( $r = 15$  cm;  $h = 48$  cm;  $v = 34$  L) where vacuum (3 kPa) is generated by a vacuum pump cooled by a closed water circuit connected to a condenser (Brat et al., unpublished results). Steam-heating liquors, generated by condensation of steam on the fruits and by exudation of some inner juice, are collected at the base of the steam-heating chamber. Aromatic liquors, generated by the instantaneous evaporation of water and volatiles, are collected after passage on a plate exchanger.

**Preparation of the Samples.** *Reference Juice (Sample 1).* Fruits (2 kg) were cut in half with a knife and introduced in a rotating pulper (Auriol, Marmande, France) equipped with a 1-mm screen. Yield: 26% juice/fresh fruit weight.

*Steam-Heated Puree (Sample 2).* Fruits (2 kg) were placed in the steam-heating chamber and heated for 10 min (final temperature, 85 °C). Steam-heated fruits were recovered and refined as above (yield: 48% puree/fresh fruit weight), whereas steam-heating liquors were discarded.

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**Vacuum-Expanded Puree.** Fruits (2 kg) were heated in the steam-heating chamber for 10 min, then introduced in the vacuum vessel at 3 kPa. Vacuum-expanded fruits (sample 3) (yield: 49% puree/fresh fruit weight) and aromatic liquors (sample 4) (yield: 10%/fresh fruit weight) were recovered. Vacuum-expanded fruits were refined further as above.

**Rind Puree (Sample 5).** After scooping out the pulp from fruits, rinds (2 kg) were steam-heated for 10 min, and then added with water (4 L). The mixture was thoroughly homogenized in a Waring Blender.

**Liquid-Liquid Extraction (LLE).** Sample (5 g) was mixed with 20 mL of distilled water; then isoamyl isobutanoate (60  $\mu$ g) was added as internal standard, followed by 30 mL of pentane/dichloromethane (2:1). The slurry was finally homogenized with a Potter-Elvehjem homogenizer for 5 min. Phase separation was achieved by centrifugation at 9000g for 5 min. The upper organic phase was recovered, dried over anhydrous sodium sulfate, and finally concentrated at 37 °C with a 25-cm Vigreux distillation column to a volume of 2 mL.

**Simultaneous Distillation-Extraction (SDE) at Atmospheric Pressure.** Sample (40 g) was mixed with 100 mL of distilled water; then isoamyl isobutanoate (130  $\mu$ g) was added as internal standard, and pH was adjusted to 7 with 1 M sodium hydroxide. The slurry was finally added to a 250-mL round-bottom flask and continuously extracted for 2 h with 15 mL of pentane/dichloromethane (2:1) at atmospheric pressure according to the procedure described by Lickens and Nickerson (1964). The organic phase was dried over anhydrous sodium sulfate and concentrated at 37 °C with a 25-cm Vigreux distillation column to a volume of 2 mL.

**Vacuum Headspace System (VHS).** Sample (50 g) was added to a 500-mL round-bottom flask; then isoamyl isobutanoate (160  $\mu$ g) was added as internal standard, and pH was adjusted to 7 with 1 M sodium hydroxide. Then a vacuum was applied for 2 h ( $\approx$ 0.1–1 kPa), fruit volatile components and water being condensed in three cooling traps (ice-water/dry ice-acetone/liquid nitrogen) (Forss and Holloway, 1967; Werkhoff et al., 1998). The aqueous condensates were combined and extracted during 30 min under stirring with 100 mL of pentane/dichloromethane (2:1). After phase separation, the organic phase was dried over anhydrous sodium sulfate and concentrated at 37 °C with a 25-cm Vigreux distillation column to a volume of 2 mL.

**Solvents and Chemicals.** The solvents (*n*-pentane and dichloromethane) were of analytical grade. Reference compounds, if available, and *n*-alkanes (C<sub>5</sub>–C<sub>22</sub>) standards were from Aldrich Chimie (Saint Quentin Fallavier, France).

**Gas Chromatography (GC) Analysis.** A Varian 3380 gas chromatograph was used with a flame ionization detector (FID), an on-column injector, a DB-Wax (column A, J&W Scientific, Folsom, CA) fused silica capillary column [30 m  $\times$  0.32 mm inside diameter (i.d.)  $\times$  0.25  $\mu$ m film] or a DB-1 (column B, J&W Scientific, Folsom, CA) fused silica capillary column (30 m  $\times$  0.32 mm i.d.  $\times$  0.25  $\mu$ m film). Oven temperature was held at 40 °C for 3 min, then increased at a rate of 3 °C/min up to 245 °C, at which it was held for 20 min. Injector temperature was raised from 20 °C to 245 °C at 180 °C/min, at which it was held for 90 min. Detector temperature was 245 °C. Hydrogen was the carrier gas at 2.0 mL/min. Injected volumes were 2  $\mu$ L of concentrated extract. Response factors were taken as 1.0 for all compounds with reference to isoamyl isobutanoate as internal standard. Linear retention indices were calculated with reference to *n*-alkanes (C<sub>5</sub>–C<sub>22</sub>). Concentrations are given as the averages of triplicate analyses.

**GC-Mass Spectrometry (MS) Analysis.** A Varian 3300 gas chromatograph coupled to a Fisons Trio-1000 quadrupole mass spectrometer with an electron ionization mode (EI) generated at 70 eV was used. The ion source temperature was 200 °C, and the filament emission current was 1 mA. The same columns as above were used for separation. Oven temperature was held at 40 °C for 3 min, then increased at a rate of 3 °C/min up to 220 °C, at which it was held for 20 min. Injector was heated from 20 °C to 245 °C at 180 °C/min. Detector temperature was 245 °C. Helium was the carrier gas at 1.1

mL/min. Electron impact mass spectra were recorded in the 40–600 amu range at 1 s/interval. Injected volumes were 1  $\mu$ L of concentrated extract.

Compounds were identified on the basis of linear retention indices on both columns (DB-Wax and DB-1) (Jennings and Shibamoto, 1980) and EI mass spectra (Wiley 275.L) from the literature or from authentic standard compounds.

**Sensory Evaluation.** A sensory evaluation was performed by 12 trained panelists with previous experience in sensory evaluation of tropical fruit odors. Assessors were asked to smell three samples [reference juice (sample 1), steam-heated puree (sample 2), and vacuum-expanded puree (sample 3) + 10% aromatic liquors (sample 4)] randomly presented in black coded glasses covered with Petri dishes. Panelists were asked to give a value (from 0 to 10) for each sample, according to an intensity scale of fresh fruity note. Statistical analysis was performed using the Fizz software (Biosystems, Dijon, France). Distribution normality was tested by the Kolmogorov and Smirnov procedure. Analysis of variance (ANOVA) showed differences among samples and statistical data obtained with this test ranged to 7.42 ( $\pm$  2.02), 5.25 ( $\pm$  2.67), and 4.17 ( $\pm$  2.17), respectively, for samples 1, 2, and sample 3 added with of 10% sample 4. Then, a least significant difference (LSD) test was performed, showing differences among means at the 5% levels (LSD = 1.91). A Friedman test (1937), after conversion of notes in ranks (sample 1, 31; sample 2, 24; sample 3 added with 10% of sample 4, 17), was also used to class products according to the sum of ranks after calculation of the limit difference (9.60) at the levels of 5%.

## RESULTS AND DISCUSSION

**Comparison of Isolation Techniques.** Because our objective was, in addition to characterizing the aroma profile of a puree from vacuum-expanded purple passion fruits, to analyze quantitatively the aroma components distribution along the process, we first compared three techniques for isolation of aroma compounds from the reference juice (sample 1): LLE, SDE, and VHS. Results are given in Table 1. Among the 54 compounds identified, whatever the technique used, esters, that is, hexyl hexanoate, hexyl butanoate, ethyl butanoate, (*Z*)-3-hexenyl hexanoate, (*Z*)-3-hexenyl butanoate, and ethyl hexanoate, were highest in concentration, as already observed by Werkhoff et al. (1998) in the yellow passion fruit (*P. edulis* f. *flavicarpa*) juice. For most compounds, LLE and SDE gave similar results except for some esters, ethyl butanoate, butyl butanoate, ethyl hexanoate, propyl hexanoate, and monoterpenes, limonene, (*Z*)-ocimene, and  $\alpha$ -pinene, which were extracted more efficiently by LLE. In LLE extract limonene concentration was 30 $\times$  and 200 $\times$  per SDE and VHS, respectively, and represented  $\approx$ 9% of the total aroma components identified vs 0.2% by VHS, as mentioned by Werkhoff et al. (1998). VHS gave much lower concentrations of volatiles. A comparative study was also conducted on other samples (data not shown), which showed similar trends. Therefore LLE with pentane/dichloromethane (2:1) was preferred for the following studies.

**Fate of Aroma Compounds along the Flash Vacuum-Expansion Process.** Because the flash vacuum-expansion process includes two consecutive steps, steam heating then vacuum-expansion, we analyzed separately aroma compounds from a steam-heated puree (sample 2), a steam-heated then vacuum-expanded puree (sample 3) and its aromatic liquors (sample 4), and a rind puree (sample 5), in comparison with a reference single-strength juice (sample 1). Results are given in Table 2.

**Table 1. Aroma Components Recovered from Single-Strength Purple Passion Fruit Juice (Sample 1) by Three Extraction Techniques**

component	concentration ( $\mu\text{g}/100\text{ g}$ )					reliability of identification <sup>c</sup>
	LRI <sup>a</sup>	LRI <sup>b</sup>	LLE	SDE	VHS	
hexanal	780	780	20	40	5	1
ethyl butanoate	790	784	980	440	470	1
4-hydroxy-4-methyl-2-pentanone	816		40	5	tr <sup>d</sup>	2
(Z)-3-hexen-1-ol	843	847	50	60	20	2
hexan-1-ol	860	858	70	90	40	1
heptan-2-one	871	872	50	10	10	2
heptan-2-ol	887	888	10	10	10	1
methyl hexanoate	904	906	tr	5	tr	2
ethyl 3-hydroxybutanoate	912	947	50	10	5	1
benzaldehyde	926	942	10	5	30	2
$\alpha$ -pinene	928	954	10	tr	tr	1
camphene	938	938	tr	tr	tr	1
butyl butanoate	982	983	140	60	60	1
ethyl hexanoate	985	987	270	140	130	1
(Z)-3-hexenyl acetate	989	987	90	60	60	1
hexyl acetate	997	1024	70	40	30	1
phenylacetaldehyde	998	1024	10	40	10	2
<i>p</i> -cymene	1005	1020	10	60	0	1
butyl 2-methyl-butanoate	1013	1022	10	20	tr	2
limonene	1015	1030	1070	30	5	1
isopropyl hexanoate	1026	1035	20	5	tr	2
( <i>E</i> )-ocimene	1029	1038	30	5	5	1
methyl 3-hydroxyhexanoate	1031		5	10	5	2
octan-1-ol	1061	1068	20	30	tr	2
(Z)-linalool oxide	1068	1068	10	10	tr	2
$\alpha$ -terpinolene	1074	1079	tr	tr	tr	1
propyl hexanoate	1080	1085	140	50	20	2
linalool	1084	1095	60	80	20	1
heptyl acetate	1085	1095	10	5	10	2
$\beta$ -farnesene	1104	1144	20	20	20	1
phenyl acetate	1166	1170	40	20	10	2
(Z)-3-hexenyl butanoate	1169	1175	600	620	460	1
butyl hexanoate	1176	1176	200	410	140	2
hexyl butanoate	1178	1181	1740	1650	1150	2
ethyl octanoate	1183	1181	20	20	50	2
octyl acetate	1195	1193	10	10	5	2
2-heptyl butanoate	1199	1194	400	430	230	2
pentyl hexanoate	1200		50	20	10	1
nerol	1207	1234	tr	5	tr	1
geraniol	1236	1243	50	40	10	1
heptyl butanoate	1276	1275	30	20	10	2
$\alpha$ -terpineol	1287	1289	260	300	250	2
phenylmethyl butanoate	1307		120	100	80	2
ethyl 3-hydroxyhexanoate	1332	1340	40	10	10	1
9-decen-1-yl acetate	1359	1383	220	160	90	2
(Z)-3-hexenyl hexanoate	1362	1370	810	950	530	2
hexyl hexanoate	1370	1371	2120	2550	1100	1
octyl butanoate	1374	1373	360	420	190	2
2-heptyl hexanoate	1388		510	580	220	2
pentyl octanoate	1393		70	110	40	2
isopentyl octanoate	1395		40	10	10	2
$\beta$ -ionone	1442	1474	370	440	260	1
heptyl hexanoate	1459		80	10	5	2
hexyl octanoate	1555	1564	240	320	80	2
total concentration ( $\mu\text{g}/100\text{ g}$ )			11745	10485	5905	

<sup>a</sup> Linear retention index from DB-1. <sup>b</sup> Linear retention index from literature. <sup>c</sup> Key for reliability of identification: 1, linear retention index and mass spectrum of reference compounds; 2, linear retention index and mass spectrum similar to published data. <sup>d</sup> tr = traces (<5  $\mu\text{g}/100\text{ g}$ ).

When compared with the reference juice (sample 1), the steam-heated puree (sample 2) exhibited higher concentrations of aroma compounds, mainly esters, but with a different ordering, that is, hexyl hexanoate, 2-heptyl hexanoate, hexyl butanoate, 2-heptyl butanoate, and octyl butanoate. Sample 2 also showed much higher concentrations of alcohols, in particular hexan-1-ol and (Z)-3-hexen-1-ol, and in monoterpene alcohols, linalool, geraniol, and nerol. Some compounds were found at similar concentrations, for example, the monoterpenes (except heat-generated *p*-cymene), some esters, that is, ethyl hexanoate, butyl hexanoate, hexyl

butanoate, hexyl hexanoate. Analysis of volatiles from a rind puree (sample 5) showed a particular richness in limonene, and also in ethyl-3-hydroxyhexanoate, (Z)-3-hexenyl butanoate, and in terpene alcohols, nerol, geraniol, and linalool. Thus, enrichment of the steam-heated puree in certain esters and terpene alcohols as compared with the reference juice can be partly explained by incorporation of some rind in the puree; indeed, the steam-heating treatment softened the outer portion of the fruit and part of the mesocarp was recovered after refining in the puree. Also some esters decreased after steam heating, namely ethyl butanoate,

**Table 2. Aroma Components Concentrations Obtained by LLE Method**

		sample				
		1	2	3	4	5
hydrocarbons	$\alpha$ -pinene	10	5	10	5	210
	camphene	0	5	0	0	0
	<i>p</i> -cymene	10	50	50	6	80
	limonene	1060	910	1740	30	2100
	( <i>E</i> )-ocimene	30	40	90	10	50
	$\alpha$ -terpinolene	0	10	10	0	30
	$\beta$ -farnesene	20	60	50	10	30
aldehydes	hexanal	20	20	5	0	30
	benzaldehyde	10	10	10	30	140
	phenylacetaldehyde	10	30	20	40	90
ketones	4-hydroxy-4-methyl-2-pentanone	40	60	70	0	0
	heptan-2-one	50	60	70	90	60
	$\beta$ -ionone	370	110	220	20	110
alcohols	( <i>Z</i> )-3-hexen-1-ol	50	290	120	200	30
	hexan-1-ol	70	1330	570	550	230
	heptan-2-ol	10	60	40	130	60
	octan-1-ol	20	140	80	50	50
terpenols	( <i>Z</i> )-linalool oxide	10	10	20	10	20
	linalool	60	610	180	690	240
	nerol	0	40	10	40	520
	geraniol	60	190	120	150	370
	$\alpha$ -terpineol	260	200	110	90	100
esters	ethyl butanoate	970	520	70	610	0
	methyl hexanoate	0	10	10	0	0
	ethyl 3-hydroxybutanoate	50	140	130	10	30
	butyl butanoate	140	60	20	20	80
	ethyl hexanoate	270	240	70	70	130
	( <i>Z</i> )-3-hexenyl acetate	90	90	70	60	100
	hexyl acetate	70	110	50	30	270
	butyl 2-methylbutanoate	10	70	20	10	0
	isopropyl hexanoate	20	100	20	10	60
	methyl 3-hydroxyhexanoate	5	30	10	5	20
	propyl hexanoate	140	260	290	110	110
	heptyl acetate	10	20	10	20	100
	phenyl acetate	40	170	80	170	130
	( <i>Z</i> )-3-hexenyl butanoate	600	250	120	40	920
	butyl hexanoate	200	150	110	5	0
	hexyl butanoate	1740	1520	1010	50	80
	ethyl octanoate	30	40	30	10	100
	octyl acetate	10	30	30	0	20
	2-heptyl butanoate	400	1070	630	20	160
	pentyl hexanoate	50	220	170	10	260
	heptyl butanoate	30	40	30	0	0
	phenylmethyl butanoate	130	20	20	10	140
	ethyl 3-hydroxyhexanoate	40	130	100	0	1840
	9-decen-1-yl acetate	220	80	90	0	0
	( <i>Z</i> )-3-hexenyl hexanoate	820	520	470	10	80
	hexyl hexanoate	2120	2540	2580	30	160
	octyl butanoate	360	570	550	20	60
	2-heptyl hexanoate	510	1750	1280	40	200
	pentyl octanoate	70	130	110	20	30
	isopentyl octanoate	40	80	60	0	90
heptyl hexanoate	80	80	50	10	270	
hexyl octanoate	240	550	540	5	140	
total concentration ( $\mu\text{g}/100\text{ g}$ )		11745	15850	12495	3650	10080

butyl butanoate, (*Z*)-3-hexenyl butanoate, phenylmethyl butanoate, decenyl acetate, and (*Z*)-3-hexenyl hexanoate. Because yields were 26% juice/fresh fruit weight and 48% puree/fresh fruit weight for the reference juice and the steam-heated puree, respectively, the decrease of esters absent or weakly concentrated in the rind, ethyl butanoate, decenyl acetate, and (*Z*)-3-hexenyl hexanoate, could be explained by a 2-fold dilution of the juice by the rind. Conversely, this cannot explain the severe decrease of (*Z*)-3-hexenyl butanoate in which the rind puree is rich and also the concomitant large increase of (*Z*)-3-hexenyl [not observed by Engel and Tressl (1983) in SDE extracts of native passion fruit juice (pH 3.0, 2 h)]. Furthermore, short-chain esters

seemed to be more sensitive than long-chain esters. Behavior of geraniol and  $\alpha$ -terpineol resulted from the 2-fold dilution, whereas the large enrichment in linalool could arise from acid hydrolysis of glycosylated precursors of linalool, in which passion fruit juice is particularly rich (Engel and Tressl, 1983; Chassagne et al., 1996). It could also have been generated by the heat treatment in the acidic condition of 3,7-dimethyloct-1-ene-3,7-diol, a precursor found in the water-soluble fraction of a chloroform extract of a yellow passion pulp (Engel and Tressl, 1983).

The puree from flash vacuum-expanded fruits (sample 3) had a lower total concentration in volatiles as compared with the puree obtained from fruits simply

steam-heated (sample 2); this is due to the instantaneous evaporation occurring when heated fruits are introduced in the vacuum chamber. Aromatic liquors (sample 4) represented 10% (w/w) of the steam-heated fruits (Brat et al., unpublished results). Impoverishment is somewhat related to the boiling temperature of the considered compounds. More specifically, for volatiles of which the concentration strongly drops after vacuum-expansion vs steam heating only (e.g., linalool, ethyl butanoate) they are relatively enriched in aromatic liquors (sample 4). Behavior of other volatiles remains unclear. Taking into account the yields,  $\approx 90\%$  of volatiles present initially in sample 2 were recovered in samples 3 and 4.

**Sensory Evaluation.** Because the vacuum-expansion provoked the instantaneous evaporation of 10% of the fruit weight as aromatic liquors (sample 4), we performed a sensory evaluation of the vacuum-expanded puree (sample 3) added with this proportion of aromatic liquors in comparison with the steam-heated puree and the reference juice. Results of ANOVA at the 5% level showed that the steam-heated puree and the vacuum-expanded puree added with 10% aromatic liquors are different from and less fruity than the reference juice. The Friedman test at the 5% level ranked the steam-heated puree between the reference juice and the vacuum-expanded puree added with aromatic liquors. Chan (1993) mentioned that the flavor and aroma of passion fruit is very sensitive to heat processing; accordingly, steam heating the fruits for 10 min up to 85 °C before vacuum-expansion must have somewhat altered the delicate fruity note of the inner juice. However, as previously mentioned, we did not detect, apart from *p*-cymene, heat-generated aroma compounds. Thus, results of this sensory evaluation indicate that passion fruit puree is actually an intermediate food product having an aroma profile different from a passion fruit juice because of incorporation of some rind in the juice.

Thus, the steam-heating step of the process afforded first a puree with increased concentration of total volatiles, in particular esters, in comparison with a reference single-strength juice, because of incorporation in the juice of part of the rind. After vacuum-expansion, the puree was impoverished in volatile components because of instant evaporation of some water when steam-heated fruits are introduced in the vacuum vessel. Most of these volatiles were recovered in aromatic liquors generated by the vacuum-expansion step, and they could be added back to the vacuum-expanded puree.

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