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Discrimination between freshly made and stored reconstituted orange juice using GC Odour Profiling and aroma values

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The aroma of freshly made and stored reconstituted orange juice was analyzed by GC– MS and GC–FID. The importance of the individual compounds was evaluated by calculation of aroma values. For comparison, the same samples were evaluated by a GC-sniffing technique called GC Odour Profiling, using a panel of five assessors. Both methods showed that there were significant differences between freshly made and stored juice, but the two methods did not always show the same compounds/odours to be important. On the other hand, many similarities were seen, as ethyl butanoate, β-pinene, limonene, octanal and linalool were shown to be important by both methods. In conclusion, both methods proved to be useful for identifying important aroma compounds in orange juice and for discrimination between fresh and stored juice. © 1998. Elsevier Science Ltd. All rights reserved

INTRODUCTION

Orange juice is the most important juice product in the world. Most of the juice on the Danish market is produced from frozen concentrate and aseptically packed in Tetra Brik™ or PurePak™. In Tetra Brik the juice is normally given a shelf life of 8 months at room temperature. The sensory quality is of great importance, and several studies have shown that the sensory quality and aroma composition changes during storage (for instance, Dürr and Schobinger, 1981; Moshonas and Shaw, 1989*a*,*b*; Velez *et al*., 1993).

The traditional way to describe the importance of changes in the volatile/component composition is to calculate aroma values by dividing the concentrations by the corresponding odour thresholds (see for instance Rothe *et al*., 1972). Another way is using 'GC sniffing' (gas chromatography-olfactometry), where assessors, during GC analysis, assess the effluent at a sniffing port. This has the advantage that the importance of unknown compounds can be evaluated since knowledge of odour thresholds is not necessary. In many cases even compounds not giving peaks on the Flame Ionization Detector (FID) can be detected and quantified. Different methods for quantitation have been presented. By dilution analysis (Acree *et al*., 1984; Schieberle and Grosch, 1984) the assessor sniffs a series of dilutions of the aroma extract. The volatile compounds detected in the most dilute sample are said to be the most important ones. By the Osme method (Miranda-Lopez *et al*., 1992), the assessor rates intensity and duration of the odours using a time–intensity device.

Dilution analysis is time-consuming because each assessor has to sniff many dilutions, so most often only one or two assessors are used. By the Osme method it is possible, with the same time resources, to allow a whole panel of assessors to sniff and even make repetitions, thus obtaining more generalizable results (since more assessors are used), and enabling evaluation of reproducibility and precision using standard statistical techniques. However, the Osme method requires more training of the assessors.

In the present study, a method we have called GC Odour Profiling was used with a panel of five assessors. This method is very similar to the Osme method, except that the odour intensity is rated by a number between 1 and 5 and only the retention time for the beginning of the odour is used. Methods similar to GC Odour Profiling have been used earlier, for analysis of orange juice using one assessor (Marin *et al*., 1992), for analysis of orange lemonade using one assessor (Fischböck *et al*., 1988), for analysis of cheddar cheese using two assessors

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(Arora *et al*., 1995), for analysis of odour-active volatiles of *Pseudomonas fragi* grown in milk using three assessors (Cormier *et al*., 1991), and for analysis of lemon and lime citrus essential oils using five assessors (Chamblee and Clark, 1993).

The aim of the present investigation was to compare freshly made and stored orange juice using GC Odour Profiling and calculated aroma values, and to evaluate the ability of the two methods to show differences between the samples. For GC Odour Profiling, a panel of five assessors was used. Aroma values were calculated for those compounds that could be identified and quantified, and where information about odour thresholds was available.

MATERIALS AND METHODS

Juice

This study used commercial orange juice, reconstituted from a Brazilian concentrate (64 °Brix) of the orange cultivar 'Pera'. The reconstituted juice (11.2 °Brix) was added ascorbic acid (200 mg litre^{-1}). The juice was packed aseptically in TetraBrik (1 litre). The TetraBrik packaging material consisted of the following layers from external to internal: PE, raw paper (liquid packaging board), LDPE, aluminium foil, acid modified LDPE and PE. 'Fresh' juice was stored for less than 2 weeks at 5°C after reconstitution, while 'stored' juice was stored at 20°C for 9–12 months. Samples were kept frozen (−18°C) from the end of the storage period until analysis.

Extraction of volatile components

Orange juice (20.0 g) with 400 μl internal standard added (50 μl litre^{-1} solution of 4-methyl-1-pentanol in water) was extracted with 20 ml diethyl ether:*n*-pentane (2:1) under magnetic stirring for 30 min. After standing for 15 min, the sample was frozen at −18°C. When the water phase was frozen, the diethyl ether:*n*-pentane phase was poured off and concentrated to 100 mg by gently blowing N_2 over the surface. For GC Odour Profiling, eight extracts were combined and concentrated to 100 mg.

Gas chromatograhy–mass spectrometry (GC–MS)

GC–MS was performed on a fresh and a stored juice sample using an HP5890 Series II gas chromatograph coupled to a Jeol JMS-AX505W mass spectrometer (Jeol Ltd, Tokyo, Japan). A DB-WAX column, 30 m×0.25 mm, 0.25 μm film thickness (J&W Scientific) was used, and the temperature program was: 40°C for 10 min, 40–230°C at 3°C min^{-1}, 230°C for 10 min, 230–240°C at 3°C min^{-1}, 240°C for 120 min. The head pressure was 70 kPa, and the injection temperature was 230°C. Split injection was applied (split ratio 1:10). The GC–MS interface line was maintained at 250°C. The spectrometer was operated at resolution 500, mass range 33–500, repetition rate 1.0 scan s^{-1}. The ion source was run in EI mode at 200–250°C, 70 eV ionization energy and 300 Ma trap current. Data processing was carried out with Jeol's 'Complement' software, which includes search facilities and a NIST-library.

Identification and quantitation

Fresh and stored juice was analyzed in triplicate by GC–FID using the same GC conditions as for the GC–MS. The peaks were identified by comparison with GC–MS data and by running solutions of reference compounds.

For quantitation, a dearomatized orange juice was prepared by vacuum distillation on a Buchi Rotavapor R-134 (Buchi, Switzerland). Portions of 1 litre juice were evaporated at approximately 30 mm Hg and 35°C until the volume was reduced to 50%, and the concentrate was then added an equivalent amount of water. By GC analysis the dearomatized juice was found to contain only a minimum of volatile compounds. The following reference compounds (5 mg litre^{-1}) were added to the dearomatized orange juice: ethyl acetate, 2-butanone, α-pinene, ethyl butanoate, 2-methyl-3-buten-2-ol, hexanal, (+)-3-carene, β-myrcene, octanal, nonanal, furfural, linalool, octanol, 5-methyl-2-furfural, 4-terpineol, butanoic acid, α-terpineol. This juice base was diluted with dearomatized juice to the following concentrations of added standards: 2.5, 1.0, 0.1 and 0.01 mg litre^{-1}. A separate quantitation was carried out for limonene, following the same procedure, except that the concentrations of added limonene were 200, 100, 50 and 10 ml litre^{-1}.

The juice bases were analyzed by GC-FID using the same conditions as above. The analyses were done in triplicate. Linear regressions between concentrations of the reference compounds and relative peak areas were used to determine concentrations of the volatile compounds in the orange juice. Approximate quantitation was carried out for other compounds assuming that compounds with similar chemical properties were extracted equally efficiently. From the concentrations obtained, aroma values (Rothe *et al*., 1972) were calculated using detection thresholds in water from the literature.

GC Odour Profiling

The extracts were analyzed on a 5890A GC equipped with an HP 9000 Chem Station. The conditions were as follows: column, HP-Innowax crosslinked PEG, 30 m × 0.25 mm, 0.25μm film thickness (J & W Scientific); injector, split ratio 1:10, temperature 250°C; detector, flame ionization, temperature 250°C; carrier; gas, helium; column flow, 1 ml min^{-1} pressure, constantly 65 kPa; the oven temperature program was the same as for GC–MS. Two microlitre samples of the extract were injected.

The column outlet was split into two lines (ratio approximately 1:1), one leading to the detector and the other leading to a sniffing port (olfactory detector outlet, OD-1, SGE, Ringwood, Australia). In the sniffing port, the effluent was mixed with humidified air.

Five trained assessors judged the odour intensity at the GC-sniffing port using a scale from 1 to 5 and described the odours. No odour descriptions were given in advance. One assessor sniffed for 40 min, then another assessor took over and sniffed the next 40 min. On the following day the procedure was repeated, except that the assessors sniffed the opposite part of the chromatogram.

In the following calculations the intensity of odours not detected by an assessor are set to 0, i.e. not treated as missing values. In other words, it is assumed that when an assessor did not detect an odour, this was not an error but was due to the fact that the concentration was below the assessor's threshold.

Statistics

Differences in concentration of odour compounds and differences in odour intensities were tested for significance using Analysis of Variance.

RESULTS AND DISCUSSION

Identification and quantitation

Table 1 lists volatile compounds identified in the juice samples (and a few unidentified compounds that corresponded to odours in the GC Odour Profiling). The quantified volatile compounds were tested for difference between concentration in the fresh and the stored juice. Significant differences ($P < 0.05$) were found for most of the compounds (those having low Coefficients of Variation, see Table 1). In general, concentrations were highest in the fresh juice except for acetic acid, β-terpineol and α-terpineol. Even though the difference was significant for acetic acid, it was small. The higher concentration of α- and β-terpineol in the stored juice would be expected, since these are degradation products of limonene (Clark and Chamblee, 1992).

For those volatile compounds that were quantified, the concentrations were within the range found by Shaw *et al.* (1993) and Nisperos-Carriedo and Shaw (1990) in juices reconstituted from concentrate. One exception was, however, octanal where the concentration in the present study was found to be approximately 10-times higher. Another was limonene, where the concentration was found to be relatively low. Cis-3-hexenol, an important contributor to the green, leafy top-note (Nisperos-Carriedo and Shaw, 1990) was not found in the present study.

It is seen (Table 2) that of the 21 quantified compounds, 11 had aroma values higher than 1, and of these nine exhibited significant difference (Table 1) between fresh and stored juice (all except α-terpineol having highest values in the fresh juice). In fresh juice the following compounds were the most important (by decreasing aroma value): limonene, octanal, nonanal, linalool, α-pinene, β-myrcene, ethyl butanoate and hexanal. In stored juice the most important compounds were: limonene, β-myrcene, linalool, α-pinene and α-terpineol.

It should, however, be noted that the average odour thresholds in Table 2 are based on different numbers of determinations, and, therefore, have very different reliabilities. This is especially important for β-pinene, octanol, 4-terpineol and α- terpineol, since there is only one determination for each compound, and the concentration is close to the threshold (aroma value close to 1). Conclusions about these compounds are very uncertain. For the remainder of the compounds, the thresholds are based on more determinations, or the aroma values are far from 1. Conclusions about whether these compounds are important or not are therefore more certain, though the exact ranking of the aroma values should be accepted with some reservation.

GC Odour profiling

During the GC Odour Profiling, 44 different odours were detected in the fresh juice and 43 in the stored juice. In total, 68 different odours were detected in the two juices. However, the agreement between assessors upon the existence of individual odours varied much. Acetic acid in the stored juice was the only odour detected by all five assessors. Compounds detected by four assessors were ethyl butanoate, β-pinene, limonene, octanal and acetic acid in the fresh juice, and limonene in the stored juice. All other odours were detected by three assessors or fewer. All assessors detected approximately the same number of odours. This apparent disagreement between assessors is, however, to be expected since different individuals have different thresholds for a given compound. This is, in fact, utilized by other researchers, for instance, van Ruth *et al.* (1995), who simply use the number of assessors in the panel detecting a given compound as peak height when sniffing chromatograms are constructed. The same authors found by GC-sniffing of dummy samples, that detection of an odour by three or fewer out of 12 assessors could be considered as 'noise'. Assuming that the same 'signal-to-noise ratio' can be applied to the present study, it means that information about odours detected by one assessor is unreliable, and also information about odours detected by two assessors should be accepted with reservation.

Figure 1 shows the results of the GC Odour Profiling. It is seen, that there was a great difference between the odour impressions of the fresh and the stored juice. There were also rather great differences between the intensities detected by each assessor. The lowest coefficients of variation were seen for limonene (67% in fresh and 71% in stored juice), acetic acid (80% in fresh and 63 % in

Table 1. Volatile compounds identified in orange juice

Retention time	Compound	Identified by		Conc. (mg litre^{-1}) (C.V. in %)		
		GC-MS	Reference	Fresh juice	Stored juice	SD
5.5	ethyl acetate	x	x	0.09 (13)	0.00 (–)	*
5.8	2-butanone	x	x	16.71 (7)	0.64 (13)	*
6.9	benzene/ethanol	x	x[a]			
8.3	2-pentanone	x	x	0.00 (–)	0.00 (–)	
10.6	α-pinene	x	x	0.45 (4)	0.19 (4)	*
11.4	ethyl butanoate		x	0.03 (72)	0.00 (–)	
12.4	2-methyl-3-buten-2-ol	x	x	0.13 (7)	0.00 (–)	*
14.4	hexanal	x	x	0.07 (71)	0.00 (–)	
15.5	β-pinene		x	0.21(3)	0.00 (–)	*
18.6	3-carene	x	x	0.14 (2)	0.09 (2)	*
19.8	β-myrcene	x	x	1.78 (3)	1.15 (2)	*
22.0	limonene	x	x	88.9 (1)	55.47 (2)	*
24.5	γ-terpinene	x	χ			
27.0	octanal	x	x	2.31(3)	0.00 (–)	*
28.9	3-methyl-2-buten-1-ol	x	x			
32.5	nonanal	x	x	0.42 (10)	0.00 (–)	*
35.1	acetic acid	x	x	0.38 (6)	0.42 (5)	*
35.7	furfural	x	x	0.21 (78)	0.00 (–)	
37.5	decanal	(t)	x			
39.0	m/z 189(15), 161(19), 113(54), 112(60), 71(88), 57(100)					
39.3	2,3-butandiol	x	x			
39.8	linalool	x	x	0.58 (0)	0.25 (4)	*
40.3	1-octanol	x	x	0.05 (7)	0.04 (3)	*
40.7	5-methyl-2-furfural	x	x	0.05 (73)	0.00 (–)	
41.7	β-caryophyllene	x	χ			
42.2	4-terpineol	x	x	0.37 (4)	0.25 (14)	*
43.1	butanoic acid	x	x	0.07 (76)	0.00 (–)	
43.4	β-terpineol	x	x	0.07 (18)	0.13 (6)	*
44.0	β-selinene	x				
45.4	γ-selinene	x				
45.9	a sesquiterpene	(t)				
46.2	α-terpineol	x	x	0.33 (2)	1.15 (7)	*
46.4	β-cubebene	x				
47.1	valencene	x	x			
47.3	a sesquiterpene[b]	x				
47.6	carvone	x	x			
47.8	m/z 204(82), 189(52), 161(93). 119(79), 107(100), 93(75)					
48.2	a sesquiterpene	(t)				
48.6	a cadinene	x				
48.9	1-decanol		x			
50.2	4-methyl pentanoic acid	x				
50.5	m/z 133(14), 100(100), 82(17), 72(56), 55(58)					
51.8	citral	(t)				
52.3	2-methyl-2-butenoic acid (+artifact)	(t)				

Bases of identification, quantitations (average of triplicate determinations), Coefficients of Variation, and test of significance of difference ($P < 0.05$) between juice types are shown. Unidentified peaks are included if they correspond to an odour in the GC Odour Profiling.

[a]Ethanol.

[b]1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methylethenyl)-naphthalene.

(t), tentative identification by GC–MS.

stored juice), and ethyl butanoate (95% in fresh and 91% in stored juice). Even though these values were much higher than what was seen for the GC–FID data (Table 1), significant differences were obtained for 11 odours, and the variation could probably be reduced by more training of the assessors.

Compounds having higher intensity in the fresh juice were ethyl butanoate, β-pinene, octanal and unidentified compounds with retention times 29.1 min (no peak by GC–FID or GC–MS, only detected by one assessor), 47.3 min (a sesquiterpene tentatively identified as 1,2,3,4,4a,5,6,8a-octahydro-4a,8-dimethyl-2-(1-methyl-ethenyl)-naphthalene), 48.2 min (a sesquiterpene, only detected by one assessor) and 51.8 min (probably citral). The intensity of the unidentified odours at 34.6 min, 38.3 min, 53.0 min and 53.7 min were highest for the stored juice (no peaks by GC-FID or GC-MS). For odours where no peaks were found, the compounds

Table 2. Odour detection thresholds in water and air, and aroma values of quantified compounds

Compound	Thresholds in water (mg litre^{-1})			Thresholds in air (mg m^{-3})	Aroma values	
	van Gemert and Nettenbreijer, 1977	Other sources	Average		Fresh juice	Stored juice
Ethyl acetate	0.3–5 (4)	0.5–5 (3)[b,d,e]	2.9	10	0.03	0
2-Butanone	—	50[a]	50	23	0.33	0.01
2-Pentanone	—	0.05[b]	0.05	5.5	0	0
α-Pinene	0.0025		0.0025	3.9	180	76
Ethyl butanoate	0.001	0.000005[b]	0.0005	0.11	60	0
2-Methyl-3-buten-1-ol	—		—	—	—	—
Hexanal	0.005–0.02 (4)	0.005–0.05 (3)[a,b,e]	0.022	0.058	3.2	0
β-Pinene	0.14		0.14	—	1.5	0
3-Carene	—		—	—	—	—
β-Myrcene	0.013	0.015[c]	0.014	—	130	82
Limonene	0.004–0.010 (2)		0.007	2.5	13,000	7,900
Octanal	0.0007		0.0007	0.007	3300	0
Nonanal	0.001	0.001[g]	0.001	0.013	420	0
Acetic acid	0.007–200 (3)	50[b]	69	0.36	0.01	0.01
Linalool	0.006	0.0005[b]	0.0033	0.35	190	76
Octanol	0.13		0.13	0.032	0.38	0.31
5-Methyl-2-furfural	—		—	—	—	—
4-Terpineol	0.34		0.34	—	1.1	0.71
Butanoic acid	0.05–40 (4)	0.5–7 (3)[a,b,f]	9.2	0.014	0.01	0
β-Terpineol	—		—	—	—	—
α-Terpineol	0.35		0.35	0.24	0.94	3.3

Thresholds in water are mainly from van Gemert and Nettenbreijer (1977), but supplied with data from Fazzalari (1978) and from other (primary) sources (*b–g*, see below). Minimum and maximum are shown with numbers of determinations in parentheses (if more than one). Thresholds in air are from Devos *et al.* (1990). Aroma values are concentrations divided by average thresholds in water. [a]Fazzalari, 1978; [b]Larsen and Poll, (1992); [c]Teranishi *et al.* (1991); [d]Mulders (1973); [e]Rothe *et al.* (1972); [f]Pyysalo *et al.* (1977); [g]Seifert *et al.* (1975).

responsible must be present in very small amounts and have correspondingly low odour thresholds.

It should be noted that during sniffing of standards, linalool and octanol were not properly separated by the GC-system. However, since the calculated aroma values in the samples were more than 200 times higher for linalool than for octanol, the odour signal is assumed to be totally dominated by linalool.

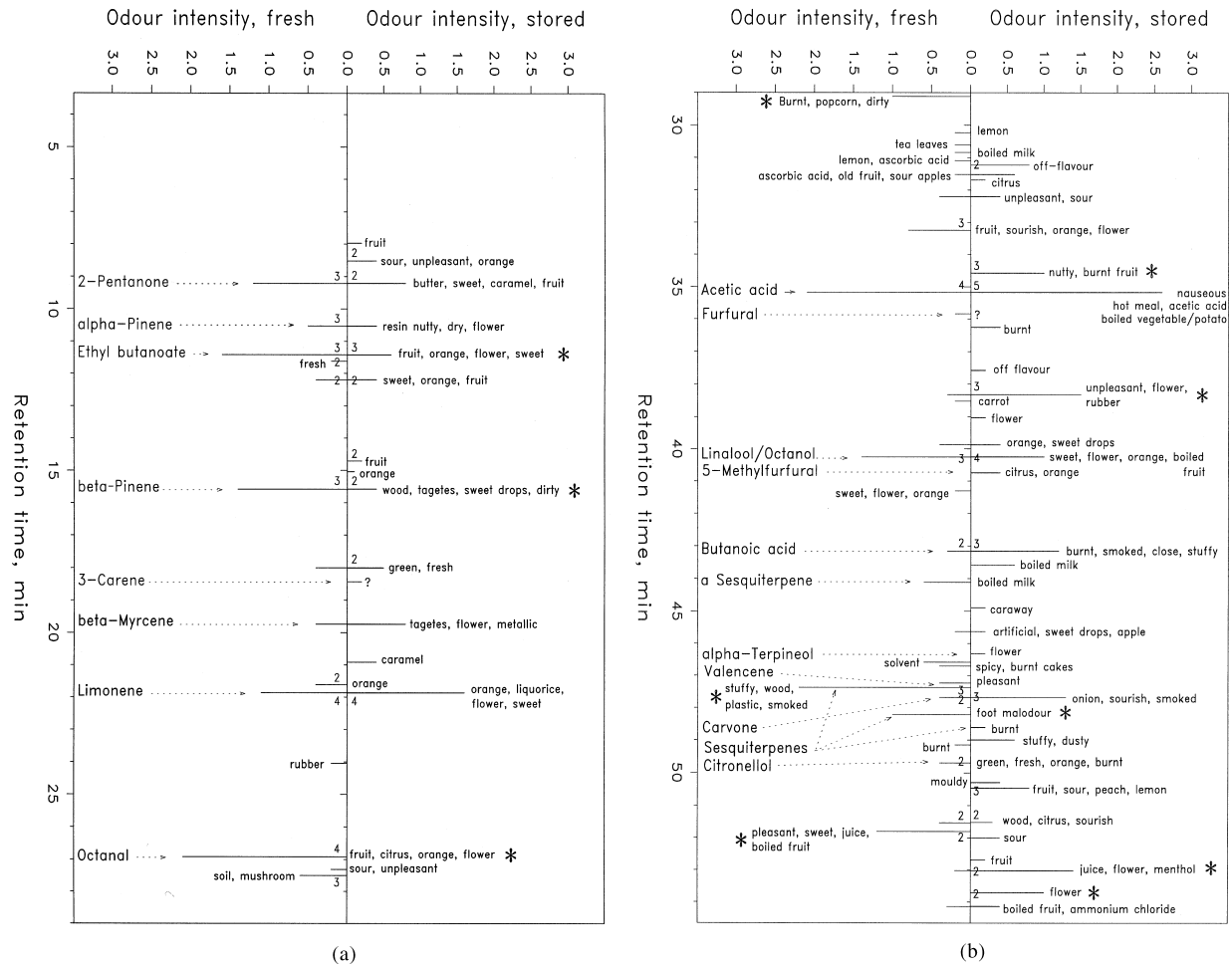
The most odour-active compounds in the fresh juice were, according to the GC odour profile (in decreasing order): a sesquiterpene (47.3 min, tentatively identified as 1,2,3,44a,5,6,8a-octahydro-4a,8-dimethyl-2 methylethenyl)-naphthalene), octanal, acetic acid, ethyl butanoate, β-pinene, linalool/octanol, 2-pentanone, citral (tentatively identified, 51.8 min), limonene and a sesquiterpene (48.2 min). For the stored juice, the most odour-active compounds were acetic acid, limonene, unidentified compounds with retention times 38.3 and 53.0 min, carvone, butanoic acid and linalool/octanol.

Among the 15 most odour-active compounds, found in orange juice by dilution analysis, Marin *et al.* (1992) identified: citral, linalool, vanillin, ethyl 2-methyl butanoate, ethyl butanoate and limonene. This is rather different from what was found in the present study. The reason for the differences is probably that the juice used by Marin *et al.* was not made from concentrate as were the ones presented here. Marin *et al.* found that limonene only had a trace odour activity, while we found that limonene plays a more important role. In the fresh juice the activity of ethyl butanoate was similar to what Marin *et al.* found.

Overall comparisons

When aroma values (Table 2) are compared with sniffing data (Fig. 1), similarities are seen. Both methods show that, during storage, concentrations of β-pinene and octanal decrease significantly. Also α-pinene, ethyl butanoate and linalool exhibit decreases, but they are not significant in either method. Similarly both methods indicate that acetic acid and α-terpineol increase during storage, but the changes are not significant in the sniffing data. In the sniffing data another eight odours exhibited significant differences between fresh and stored juice and, of these, only one was detected (and tentatively identified as a sesquiterpene) by GC-MS/GC-FID. For the remainder, no corresponding peaks occurred.

Both methods show ethyl butanoate, limonene, octanal and linalool to be important. However, it is striking that acetic acid and butanoic acid are very prominent during sniffing, even though their aroma values are practically zero. The explanation for this is that the evaluation of an aroma compound's importance is based on threshold in air when sniffing is used (since all compounds are evaporated in the GC), while it is based on threshold in water when aroma values are used. The partition coefficient between water and air, or the



(a) (b)

Fig. 1. Intensity (average) and description of odours detected during sniffing of fresh and stored orange juice. *Indicates significant difference ($P < 0.05$) between the intensity for the fresh and the stored juice (LSD = 1.0). When an odour was detected by more than one assessor the number of agreeing assessors is shown at the base of the corresponding bar.

hydrophobicity, describe the relation between threshold in water and air. Acetic and butanoic acid are very hydrophilic and, therefore, have much higher thresholds in water than most other compounds identified. This is not the case for thresholds in air (Table 2). The use of GC-sniffing will therefore tend to overestimate the importance of these compounds. It should, however, be noted that this is only a problem when extraction methods are used. In headspace methods the partition coefficient will influence the evaporation from the sample, and thereby the amounts of volatiles trapped and sniffed.

In addition to the odours already mentioned, a rather high number of less potent odours were detected by sniffing but not by GC–FID/GC–MS. These are not key-odourants but may well be important for the total sensory impression of the juice.

Another type of difference between the methods is that the reproducibility is better in the GC–FID data than in the GC Odour Profiling data. Ethyl butanoate is one exception from this, since it had a significantly higher odour intensity in the fresh juice while the difference in concentration was not significant. This can be explained by ethyl butanoate's low odour threshold and low concentration. These in combination make it easy to sniff, but difficult to detect instrumentally.

In a few cases (β-myrcene, limonene and butanoic acid) differences detected by the two methods seem to be in opposite directions, but none of the differences in odour intensity were significant, and only two of the differences in aroma value were significant (β-myrcene and limonene).

CONCLUSION

In conclusion, both the GC Odour Profiling method and the calculation of aroma values proved to be useful for discrimination between fresh and stored juice and for identifying important aroma compounds in orange juice. Calculation of aroma values is more reproducible in most cases, but demands that all aroma compounds of interest are identified and quantified, and that reliable and comparable threshold values can be obtained.

GC Odour Profiling often gives a poorer reproducibility, except for compounds with low thresholds present in low concentrations. An advantage using GC Odour Profiling is that unknown compounds, and even

compounds not detectable by GC–FID/GC–MS, can be described and quantified. It is a very common experience that potent odours cannot be detected by GC–FID or GC–MS. In comparison with GC sniffing using dilution techniques, GC Odour Profiling has the advantage that, with the same time consumption, it is possible to use more assessors and thereby obtain more representative results, that can be evaluated by standard statistical techniques.

A problem that all GC-sniffing techniques have when used in combination with solvent extraction is the tendency to overestimate the importance of strongly hydrophilic compounds, but this can, to a certain degree, be overcome if partition coefficients of the compounds are known.

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