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Method for the static headspace analysis of carrot volatiles

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Abstract

A static headspace analysis/gas chromatography/mass spectrometry (SHA/GC/MS) method was developed to analyse the volatile composition of raw (seven different varieties), stored and cooked carrot samples. A total of 35 different volatile compounds were identified in carrots. Of these, trans-ocimene, 2,5-dimethyl styrene, camphor, borneol, α-santalene, α-selinene, γ-elemene and α-zingiberene in raw carrots and propanol in stored carrots were identified for the first time. Major volatile compounds identified in raw carrots were α-pinene, sabinene, myrcene, limonene, γ-terpinene, terpinolene, β-caryophyllene and γ-bisabolene. Mono-and sesquiterpenes accounted for about 97% of the total volatiles identified. Sizeable varietal differences ($p < 0.01$) were observed. Carrot volatiles did not change appreciably during the 28 day storage period at 5, 25 and 35°C, except propanol that showed exponential increases at higher temperatures. No propanol was detected in fresh raw carrots. Cooking resulted in 88.6, 93.0 and 95.5% loss in total volatiles after cooking times of 10, 20 and 30 min, respectively.

1. Introduction

Carrots are a relatively important crop in the UK, and are consumed extensively both raw and cooked. In recent years the consumption of carrots in the UK has been increasing (total output marketed in England and Wales = 460 950 and 603 560 Mt. in 95/96 and 96/97, respectively; MAFF, 1997), mainly because of their pleasant flavour, relative cheapness, colour and health interests of the consumer. Carrots are the major vegetable source of provitamin A, especially β-carotene which may modify susceptibility to western-type diseases such as atherosclerosis (COMA, 1994).

Carrots have a complex flavour. There is no single compound that accounts for a distinctively carrot-like flavour (Simon, 1985). Although there are many factors that influence carrot flavour, including non-volatile chemical constituents such as free sugars, phosphates and nitrogenous compounds (Alabran & Mabrouk, 1973), bitter compounds (Carlton, Peterson, & Tolbert, 1961), phenolic compounds (Howard, Braswell, Heymann, Lee, Pike, & Aselage, 1995; Sarkar & Phan, 1979), and organic acids (Howard et al., 1995) the characteristic flavour of carrots is mainly due to the volatile constituents which are mostly made up of terpenes and sesquiterpenes (Buttery, Seifert, Guadagni, Black, & Ling, 1968; Heatherbell & Wrolstad, 1971; Heatherbell, Wrolstad, & Libbey, 1971a,b; Lund & Bruemmer, 1992; Seifert & Buttery, 1978; Shamaila, Durance, & Girard, 1996; Simon, 1985; Simon, Peterson, & Lindsay, 1980b).

Subjective grading of carrot quality by sensory assessment can be reliable, provided that it is correctly practised. It is, however, time consuming and costly-particularly for screening large numbers of varieties for plant breeding. Earlier objective studies employed various extraction techniques to isolate carrot flavour: conventional Likens–Nickerson distillation (Buttery et al., 1968), aqueous extracts (Heatherbell et al., 1971a), on-column trapping (Heatherbell et al., 1971b). Such techniques are laborious, tedious, time-consuming and require large volumes of sample. Therefore, there is a need for a rapid, simple and effective technique to objectively measure the most influential odorants (terpenes and sesquiterpenes). Both static and dynamic headspace analysis (SHA & DHA) coupled with gas chromatography/mass spectrometry (GC/MS) have gained in popularity for analysing key odorants in foods. Although DHA is considered the more sensitive of the two techniques (Alasalvar, Quantick, & Grigor, 1997; Moshonas & Shaw, 1992), SHA maybe more attractive as a technique for routine quality control due to its operational simplicity and repeatability (Baldwin, Nisperos-Carriedo, Baker, & Scott, 1991; Lizotte &

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Shaw, 1992; Malundo, Baldwin, Moshonas, Baker, & Shewfelt, 1997; Moshonas & Shaw, 1992). This technique involves the chromatographic separation of a predetermined volume of vapour headspace above a sample held in a closed vial.

During storage, a number of microbial deteriorations may affect carrots. Moulds such as *Botrytis cinerea*, *Mycocentrospora acerina* and *Sclerotinia sclerotiorum* and soft rot bacteria such as *Erwinia carotovora* and *Pseudomonas* spp. commonly limit the storage life of carrots (Lapwood, 1981). It is possible that the volatile compounds produced by these organisms might be used as indicators of incipient spoilage through the analysis of the storage environment. Volatiles have been used as markers of microbial deteriorations in several processed foods (Eyles & Adams, 1986; Guarino & Kramer, 1969; Schafer, Peeler, Bradshaw, Hamilton, & Carver, 1982).

Few studies have been conducted on volatile changes in carrots during cooking. Simon and Lindsay (1983) found 70–85% loss of total volatiles after 11 min cooking time. In the UK it is likely that some consumers prepare carrots with longer cooking times than 11 min, and therefore as an aid to the study of consumer perception it is important to assess how further cooking could affect volatile composition.

Therefore the objectives of this study were to: (a) develop a method to identify the major volatiles in carrots using SHA, and examine how they differ between varieties; (b) define the storage life of carrots by measuring volatile changes during different storage temperatures; (c) assess how cooking time affects the volatile composition of carrots.

2. Materials and methods

2.1. Materials

Seven carrot F1 hybrid varieties (designated as 2, 3, 7, 8, 9, 10, and 11) were sown in the same location in sandy, silt soil in May and were lifted for analysis in November of the same year. β- and γ-bisabolene were obtained from Tokyo Kasei Organic Chemicals Ltd., Japan. β-farnesene was obtained from Wako Chemicals Ltd., Japan. All other chemicals were purchased from Aldrich, Fluka and Sigma Chemicals Co., UK, unless otherwise indicated.

2.2. Preparation of sample

Carrots from each variety ($\cong$ 3–5 kg) were ''topped'' and ''tailed''. The samples were then placed inside paper bags and stored at chilled condition (2–5°C) with a relative humidity of 70–90%, until analysed. Variety 2 was also stored at 25 and 35°C. All carrots were thoroughly washed with tap water and scraped (to remove the skin) before analysis.

For cooked and stored carrot volatile analysis, variety 2 was used. The carrot sample was sliced (5 mm thickness) and immersed in pre-boiling distilled water for 10, 20 and 30 min, drained and analysed. For studying the effects of storage on carrot volatiles, variety 2 was analysed immediately on arrival in the laboratory (day 0) and then equally divided into three lots so that each lot was stored at temperatures of 5, 25 and 35°C. Analysis of volatiles took place on days 1, 4, 7, 11, 14, 20, 24 and 28. Once the sample had deteriorated beyond the point of being acceptable for human consumption (i.e. severe darkening of the skin, moisture lost and alcoholic smell, etc.), the sample was discarded.

2.3. Preparation of solutions of authentic compounds

For the purpose of positive identification and quantitation, standard solutions of the terpenoids listed in Table 2 were first dissolved in methanol (to improve the solubility of the standards) at a concentration of 1000 ppm. The final concentration between 0 and 10 ppm was prepared with HPLC water. A 5 ml aliquot of mixed standard solution was then transferred into a 22 ml headspace vial and sealed for analysis.

2.4. SHA/GC/MS

Mass spectra of volatiles were obtained by a combination of a Varian Genesis Headspace Autosampler, Star 3400 CX GC and a Saturn GC/MS/MS 4D (Varian Associates Inc., CA).

A 5 g grated carrot was transferred into a 22 ml headspace vial, immediately sealed (with crimp top aluminium caps) and analysed for headsapace volatiles composition. Triplicate analyses was performed for each sample. Optimum SHA conditions were developed as shown in Table 1.

A Varian Star 3400 CX GC was used with a high resolution gas chromatography column (DB-5MS; 30 m $\times$ 0.25 mm i.d $\times$ 0.25 μm film thickness; J & W Scientific, Folsom, CA) operated with ultrahigh purity helium with a carrier gas flow rate of 1 ml/min. Each sample was injected in the splitless mode (240°C injection temperature; 70 s valve delay). The column temperature was programmed

Table 1
The SHA conditions developed for carrot volatiles analysis

Plate temperature	85°C
Sample equilibrium time	30 min
Mixing time	5 min
Mixer power	10
Loop size	1 ml
Loop equilibrium time	0.10 min
Injection time	0.50 min
Sample loop temperature	110°C
Line temperature	110°C
Transfer line back pressure	13 psi

Table 2
Quantitative values (ppm) of volatile compounds for different varieties of raw carrot

Peak no.[a]	Compound name	Variety						
		2	3	7	8	9	10	11
3	α-Pinene	1.53 ± 0.059	1.96 ± 0.074	1.16 ± 0.052	1.23 ± 0.016	1.45 ± 0.058	0.153 ± 0.015	0.111 ± 0.010
4	Camphene	0.079 ± 0.008	0.138 ± 0.015	0.043 ± 0.003	0.052 ± 0.006	0.065 ± 0.013	0.001 ± 0.001	0.003 ± 0.001
5	Sabinene	0.045 ± 0.004	0.069 ± 0.008	0.111 ± 0.004	0.067 ± 0.003	0.099 ± 0.007	2.46 ± 0.141	0.390 ± 0.025
6	β-Pinene	0.085 ± 0.008	0.108 ± 0.011	0.042 ± 0.002	0.028 ± 0.005	0.128 ± 0.012	0.120 ± 0.011	0.222 ± 0.015
7	Myrcene	0.285 ± 0.044	3.64 ± 0.092	8.86 ± 0.307	0.418 ± 0.028	17.6 ± 0.435	0.099 ± 0.009	0.118 ± 0.009
8	α-Phellandrene	0.054 ± 0.005	0.034 ± 0.005	0.066 ± 0.010	0.037 ± 0.005	0.138 ± 0.011	0.076 ± 0.005	0.071 ± 0.003
9	α-Terpinene	0.003 ± 0.001	0.019 ± 0.003	0.027 ± 0.002	0.004 ± 0.001	nd[b]	0.056 ± 0.003	0.006 ± 0.002
10	*p*-Cymene	0.013 ± 0.002	0.039 ± 0.006	0.121 ± 0.007	0.046 ± 0.006	0.044 ± 0.002	0.005 ± 0.001	0.008 ± 0.002
11	Limonene	1.04 ± 0.039	0.571 ± 0.056	1.27 ± 0.081	0.247 ± 0.010	1.62 ± 0.082	0.391 ± 0.029	0.104 ± 0.010
12	*cis*-Ocimene	0.268 ± 0.035	0.007 ± 0.001	0.039 ± 0.006	0.024 ± 0.002	0.148 ± 0.007	0.069 ± 0.011	0.006 ± 0.001
13	*trans*-Ocimene	0.125 ± 0.017	0.015 ± 0.007	0.083 ± 0.008	0.011 ± 0.002	0.374 ± 0.029	0.032 ± 0.008	0.035 ± 0.008
14	γ-Terpinene	0.380 ± 0.046	1.82 ± 0.046	3.38 ± 0.131	1.10 ± 0.005	1.65 ± 0.015	0.570 ± 0.048	0.203 ± 0.012
15	Terpinolene	0.600 ± 0.031	0.898 ± 0.072	1.46 ± 0.059	0.600 ± 0.046	1.14 ± 0.027	1.59 ± 0.074	0.760 ± 0.053
16	2,5 Dimethyl styrene	0.004 ± 0.001	0.035 ± 0.003	0.025 ± 0.004	0.020 ± 0.003	0.031 ± 0.005	0.044 ± 0.005	0.009 ± 0.001
17	Undecane	0.003 ± 0.001	0.070 ± 0.008	0.003 ± 0.001	0.059 ± 0.012	nd	0.019 ± 0.002	tr[c]
18	Camphor	0.222 ± 0.012	0.147 ± 0.004	0.145 ± 0.005	0.114 ± 0.009	0.089 ± 0.002	0.090 ± 0.005	0.067 ± 0.007
20	Terpinen-4-ol	0.084 ± 0.008	nd	nd	nd	nd	0.071 ± 0.008	nd
23	Bornyl acetate	0.258 ± 0.020	0.113 ± 0.012	0.033 ± 0.003	0.012 ± 0.001	nd	nd	0.021 ± 0.004
25	Longifolene	0.001 ± 0.001	0.009 ± 0.001	nd	tr	nd	tr	0.004 ± 0.001
26	β-Caryophyllene	2.80 ± 0.040	0.865 ± 0.074	1.27 ± 0.041	0.012 ± 0.004	5.59 ± 0.130	0.718 ± 0.043	1.28 ± 0.061
28	*trans*-α-Bergamotene	0.069 ± 0.005	0.042 ± 0.005	0.074 ± 0.005	0.034 ± 0.006	0.009 ± 0.001	0.011 ± 0.000	0.087 ± 0.007
29	α-Humulene	0.101 ± 0.005	0.051 ± 0.002	0.039 ± 0.005	0.005 ± 0.000	0.230 ± 0.003	0.022 ± 0.001	0.038 ± 0.007
30	*cis*-β-Farnesene	0.128 ± 0.008	0.066 ± 0.008	0.111 ± 0.007	0.030 ± 0.004	0.012 ± 0.001	0.006 ± 0.001	0.079 ± 0.007
33	Valencene	0.021 ± 0.001	0.007 ± 0.001	0.010 ± 0.001	0.004 ± 0.001	0.035 ± 0.003	0.003 ± 0.001	0.010 ± 0.001
34	β-Bisabolene	0.112 ± 0.008	0.075 ± 0.008	0.124 ± 0.003	0.101 ± 0.015	0.084 ± 0.010	0.032 ± 0.002	0.737 ± 0.022
35	γ-Bisabolene	1.60 ± 0.099	1.20 ± 0.061	1.66 ± 0.046	1.74 ± 0.087	0.361 ± 0.039	0.700 ± 0.046	0.228 ± 0.009
	Monoterpenes	4.50 (45.45)[d]	9.32 (77.68)	16.7 (82.69)	3.87 (64.46)	24.5 (79.19)	5.62 (76.62)	2.04 (44.34)
	Sesquiterpenes	4.83 (48.78)	2.31 (19.28)	3.28 (16.29)	1.93 (32.12)	6.32 (20.43)	1.49 (20.33)	2.46 (53.55)
	Total volatiles	9.90 (100)	12.0 (100)	20.2 (100)	6.00 (100)	30.93 (100)	7.34 (100)	4.59 (100)

[a] Peak numbers correspond to the peaks in Figs. 1 and 2.
[b] nd (not detected) represents not detected compounds.
[c] tr (trace) represents concentration of less than 0.001 ppm.
[d] Numbers in brackets indicate percent of compounds to the total amount of volatiles.
Data are expressed in mean ± SD (n = 3).
Results are expressed on a fresh weight basis.

from 50 to 145°C at a rate of 3°C/min. Transfer line temperature was held at 155°C.

MS conditions were: ion source temperature; 180°C, ionization voltage. 70 eV, mass scan range, 33–350 a.m.u., electron multiplier voltage, 1750 V and scan rate, 1000 ms.

2.5. Identification and quantitation of carrot volatiles

The mass spectra were tentatively identified by comparison to reference spectra of TERPENOID and NIST 92 mass spectral database (Varian Associates, Inc., 1992). Compounds tentatively identified were confirmed by comparing their mass spectra and GC retention times to those of authentic compounds analysed in this laboratory under identical experimental conditions. Electron ionization (EI) was used. Further, chemical ionization (CI) using methane gas was also employed to aid identification. Positively identified compounds were quantified using multipoint external standard calibration curves under identical experimental and chromatographic conditions. Quantitation was performed using peak area.

2.6. Statistical analysis

Statistical significance was checked by Microsoft Excel 7 for Windows 95 using Two Sample *t*-Test, assuming equal variances.

3. Results and discussion

3.1. Volatile compounds

A representative total ion chromatograph from raw carrot (variety 2) is shown in Fig. 1. A total of 26 volatile headspace compounds were positively identified and

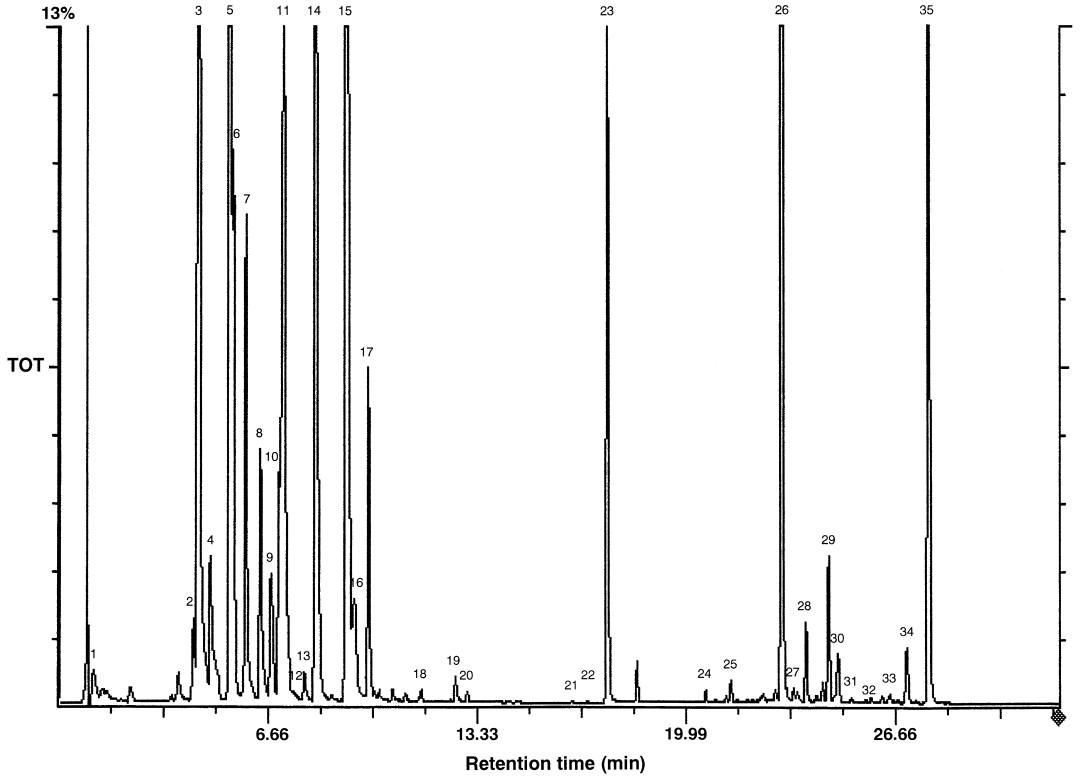

Fig. 1. Typical chromatogram of volatile compounds from raw carrots (variety 2).

quantified for different varieties of raw carrots (Table 2). Of these, trans-ocimene, 2,5-dimethyl styrene, camphor and valencene, in raw, and propanol (Fig. 2, peak no.1) in stored carrots, have not been reported previously. A further nine headspace volatiles were also tentatively identified. These were α-thujene (2), borneol (19), linalyl acetate (21), β-citronellol (22), α-santalene (24), α-selinene (27), γ-elemene (31) and α-zingiberene (32). No attempt was made to quantify them as they contributed to less than 1% of total peak area. Positively identified compounds ($>98\%$) only will be discussed in detail.

Fresh carrot volatiles mainly consist of simple monoterpenes (2–15), dimethyl substituted styrene (16), alkane (17), aromatic terpene (18), terpene alcohols (19, 20, 22), terpene acetates (21, 23) and sesquiterpenes (24–35). Mono- and sesquiterpenes accounted for about 97% of the total volatiles extracted from raw carrots. The percentages of monoterpenes were higher than sesquiterpenes in varieties 3, 7, 8, 9 and 10, whereas monoterpenes were lower than sesquiterpenes in other varieties. Such terpenes, which impart the characteristic aroma typical of carrots, are considered to be the most important volatile compounds (Buttery et al., 1968; Heatherbell & Wrolstad, 1971; Heatherbell et al., 1971a,b; Lund & Bruemmer, 1992; Seifert & Buttery, 1978; Shamaila et al., 1996; Simon et al., 1980b). It has been proposed that sabinene and particularly myrcene are responsible for notes on "green", "earthy" and "carrot top" flavours, whereas terpinolene and to a lesser extent caryophyllene, are responsible for perfumery notes in carrots (Heatherbell et al., 1971a).

Data in Table 2 show that large varietal differences exist for carrot volatiles. Total volatiles range from 30.93 to 4.59 ppm, being highest in variety 9 and lowest in variety 11. In variety 9 the most abundant volatile terpenoid, comprising about 57% of the total volatiles, was myrcene, whereas it was only 1.3% in variety 10. Although the total volatile concentration was higher in variety 9 than others, no α-terpinene, undecane, terpinen-4-ol, bornyl acetate or longifolene was detected. All detectable compounds, however, were present in variety 2. There were highly significant differences $p<0.01$) in total volatile contents among the varieties.

The precision of the technique was assessed by calculating the standard deviation (SD) for triplicate measurements as shown in Table 2. From these data the average % relative standard deviation (RSD) was calculated for two groups of measurements. Generally a reasonable level of precision (average RSD = 7.3%) was maintained for measurements above 0.01 ppm which accounted for 89% of all measurements made in Table 2. Much larger deviations (average RSD = 25%) were observed for measurements made below 0.01 ppm which indicates that this value is approaching the limits of reproducibility for the test.

Major compounds identified in all raw carrot varieties examined were α-pinene, sabinene, myrcene, limonene, γ-terpinene, terpinolene, β-caryophyllene and γ-bisabolene.

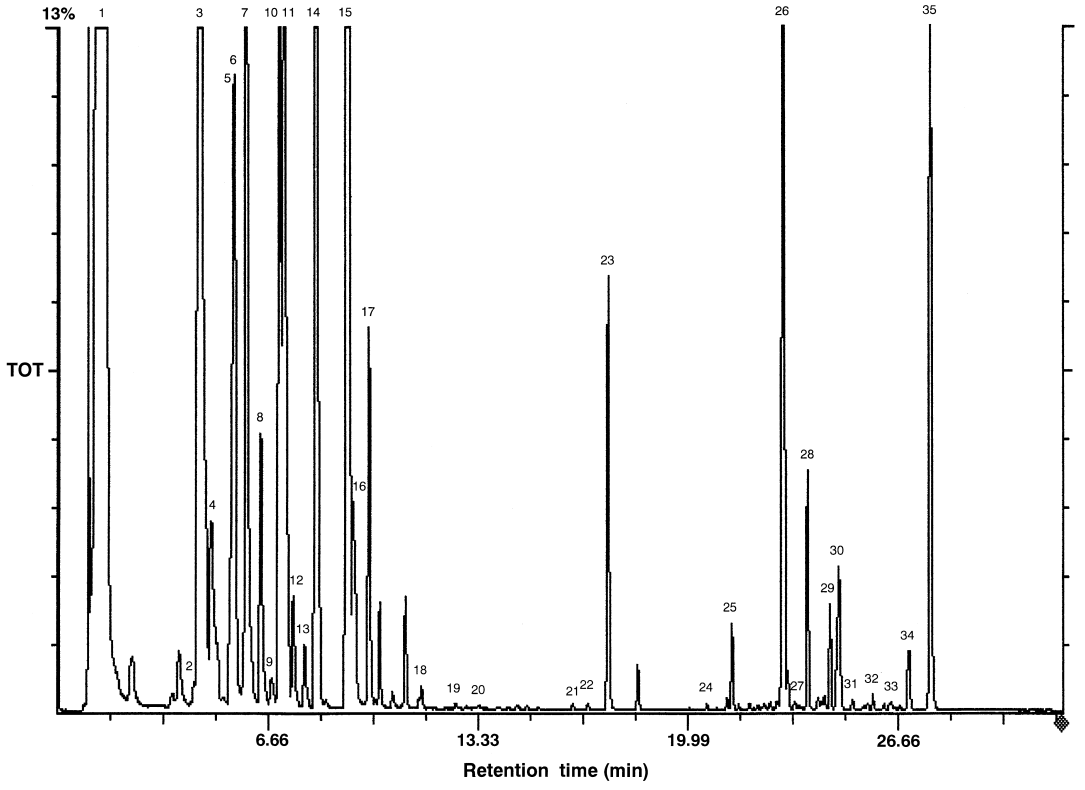

Fig. 2. Typical chromatogram of volatile compounds from stored carrots at 35°C for 7 days (variety 2).

Our findings agree well with other previous studies (Buttery et al., 1968; Heatherbell et al., 1971a,b; Howard et al., 1995; Shamaila et al., 1996; Simon et al., 1980b). Terpinolene was usually the most abundant volatile terpene reported (Buttery et al., 1968; Heatherbell & Wrolstad, 1971; Shamaila et al., 1996; Simon et al., 1980b), but caryophyllene and/or (E)-γ-bisabolene were sometimes more plentiful (Simon, 1982b). The flavour of raw carrots has been reported as largely influenced by genetic variation (Heatherbell et al., 1971a; Simon 1982a,b; Simon, Peterson, & Lindsay, 1980a). Where the effect of different soils and climates was considered, the conclusion was drawn that soils do make a difference but genotype can have greater influence (Simon, Peterson, & Lindsay, 1982).

Buttery et al., (1968), using steam distillation at atmospheric pressure for extraction and isolation of the volatiles, identified seven long-chain aldehydes which were not detected either in this study or others (Howard et al., 1995; Shamaila et al., 1996; Simon, Lindsay, & Peterson, 1980c). The production of these aldehydes is increased by heating (Buttery et al., 1968), and they can be formed from C_{18} unsaturated fatty acid auto-oxidation (Kenney, 1962); therefore, the milder static headspace extraction treatment used in this study may have limited their formation. In addition, Heatherbell et al. (1971a,b) identified six low-boiling compounds (bp < 78–80°C) in raw carrots. These compounds were also not detected either in this study or others mentioned above. The detection of low-boiling compounds is limited using the SHA technique and no attempt was made to identify these volatiles though some small broad peaks were observed at early scan numbers.

3.2. *Effect of storage on the volatiles of carrot*

Carrot volatiles did not change appreciably during 28 days of storage, except propanol (Fig. 2, peak no.1) which showed exponential increases at both 25 and 35°C. Table 3 shows the propanol changes at three different storage temperatures. Significant quantities of propanol were observed at 35°C on day 4 and at 25°C on day 14. The samples were discarded for further analysis after 7 days of storage at 35°C and 14 days of storage at 25°C, due to being visually unacceptable for human consumption (i.e. severe darkening of the skin, moisture lost and alcoholic smell etc.). Although propanol was detected in low concentrations at 5°C on day 14, it did not change appreciably on the remaining days of the experiment. The effects of the different levels of propanol on flavour quality were never assessed in detail; however, it was observed that, as the product started visually to deteriorate, an alcoholic odour was observed. A number of spoilage bacteria have been reported to produce propanol and other alcohols, including *Pseudomonas* spp. which are implicated in the spoilage of stored carrots (Ahmed & Matches, 1983). Further, it seems plausible that measuring the level of

Table 3
Propanol changes (ppm) of carrots abused at different temperatures

Storage time (day)	Storage temperatures (°C)		
	5	25	35
0	nd[a]	nd	nd
1	nd	nd	nd
4	nd	14.1 ± 1.25	265 ± 21.2
7	nd	26.8 ± 1.56	1307 ± 80.6
11	nd	141 ± 13.8	dr
14	8.85 ± 0.63	823 ± 25.6	dr
20	9.26 ± 0.25	dr[b]	dr
24	11.1 ± 0.58	dr	dr
28	11.5 ± 1.00	dr	dr

[a] nd (not detected) represents not detected compounds.
[b] dr (disregarded) represents disregarded samples due to being visually unacceptable for human consumption.
Data are express in mean ± SD (n−3).

propanol before the odour can be detected in carrot could be used as an indicator of microbiological spoilage, and therefore as a potential sensitive test of the post harvest life of the product in good condition.

Heatherbell and Wrolstad (1971) reported that immature carrots, stored for 5 weeks in polyethylene bags at 5°C in a dark ventilated room, accumulated large quantities of acetaldehyde and ethanol, which are indicative of anaerobic respiration (Amerine, 1964; Cossins & Beevers, 1963). Evidently the polyethylene bags created a reduced O_2 tension due to the respired CO_2. The concentrations of other volatiles (higher-boiling, bp > 78–80°C) did not change significantly at the end of the storage period (Heatherbell & Wrolstad, 1971). Simon (1984) reported that volatile terpenoids normally responsible for harshness in fresh carrots do not change appreciably during 120 days of storage at 2°C in air or air plus ethylene. He also could not detect any bitterness during storage in air.

3.3. Effect of cooking on the volatiles of carrot

Table 4 shows the volatiles (%) remaining after 10, 20 and 30 min cooking times. Highly significant ($p < 0.01$) volatile losses were observed during cooking. The levels of average total volatiles were reduced by 88.6, 93.0 and 95.5% from their original values (Table 2) in variety 2 after cooking times of 10, 20 and 30 min, respectively. Cooking clearly would appear to reduce the volatiles of carrots. These losses may occur through evaporation, leaching into the water, degradation during heat treatment and/or to less enzymatic activity. Simon and Lindsay (1983) found a loss of 70 to 85% of total volatile terpenoids in fresh-cooked carrots (1 cm thick) after 11 min cooking time in boiling salted water (3 min boiling and 8 min simmering). These results correlate well with our findings. Sesquiterpenes in general seem to survive the cooking process better than monoterpenes, which is in agreement with the findings of Heatherbell et al. (1971a).

Table 4
Remaining volatiles (%) of carrots on cooking time

Compound name	Cooking time (min)		
	10	20	30
α-Pinene	16.5 ± 3.03	9.03 ± 0.40	4.79 ± 0.61
Camphene	14.7 ± 1.00	5.17 ± 0.89	3.99 ± 0.48
Sabinene	11.8 ± 0.96	4.77 ± 0.79	2.21 ± 0.42
β-Pinene	9.62 ± 1.90	4.71 ± 0.65	2.86 ± 0.34
Myrcene	8.97 ± 1.83	0.44 ± 0.07	0.26 ± 0.03
α-Phellandrene	11.3 ± 0.64	8.12 ± 0.60	5.90 ± 0.03
α-Terpinene	12.8 ± 1.25	5.78 ± 0.71	1.66 ± 0.17
p-Cymene	5.65 ± 1.24	2.54 ± 0.19	1.38 ± 0.02
Limonene	10.0 ± 1.87	1.77 ± 0.39	0.88 ± 0.20
cis-Ocimene	8.67 ± 1.75	2.11 ± 0.15	0.85 ± 0.04
trans-Ocimene	8.12 ± 1.12	2.71 ± 0.39	1.39 ± 0.15
γ-Terpinene	14.5 ± 1.94	7.59 ± 0.92	3.90 ± 0.33
Terpinolene	12.6 ± 1.30	4.28 ± 0.94	2.15 ± 0.14
2,5 Dimethyl styrene	8.24 ± 1.53	2.76 ± 0.37	1.56 ± 0.32
Undecane	9.73 ± 0.47	7.09 ± 0.64	3.94 ± 0.66
Camphor	10.6 ± 1.06	6.36 ± 0.94	3.05 ± 0.23
Terpinen-4-ol	8.27 ± 0.61	5.43 ± 0.74	2.79 ± 0.25
Bornyl acetate	5.86 ± 0.79	3.54 ± 0.43	2.91 ± 0.31
Longifolene	13.8 ± 0.46	13.1 ± 1.83	9.95 ± 1.73
β-Caryophyllene	15.9 ± 1.20	15.6 ± 1.12	11.0 ± 2.20
trans-α-Bergamotene	10.3 ± 0.30	9.20 ± 0.22	6.90 ± 1.19
α-Humulene	16.9 ± 0.97	16.0 ± 0.96	12.4 ± 1.43
cis-β-Farnesene	12.8 ± 0.63	10.5 ± 1.02	7.75 ± 0.47
Valencene	14.8 ± 1.02	12.0 ± 1.12	8.18 ± 0.85
β-Bisabolene	3.69 ± 0.68	2.63 ± 0.40	1.78 ± 0.12
γ-Bisabolene	20.1 ± 1.38	18.3 ± 0.68	11.7 ± 1.01
Total volatiles[a]	11.4 ± 0.62	6.98 ± 0.14	4.47 ± 0.39

[a] Remaining average total volatiles.
Data are expressed in mean ± SD, (n = 3).

Carrots have been reported to lose about half of their high-boiling volatile terpenoids upon canning. However, canning produced an increase in lower-boiling compounds (ethanethiol, dimethyl sulfide, acetaldehyde, propanal, acetone and methanol). Dimethyl sulfide and ethanethiol, due to their very low odour thresholds (ppb range), are considered important contributors to canned carrot flavour (Heatherbell et al., 1971a).

4. Conclusions

The combined SHA/GC/MS technique has certain advantages over other types of technique used in carrot volatiles extraction (described in introduction). Firstly, it can identify important volatile terpenoids in carrots and could be used to provide data on carrot flavour. Secondly, this technique has potential to be used for quality control due to its simple, reliable and rapid operation. Finally, it also incurs low risk of artefact formation, requires less sample for extraction as compared to other techniques, and involves minimal sample preparation.

Storage temperature is very important to the quality of the final product. To reduce quality loss during storage, carrots have to be stored at a low temperature (0–5°C) with a relative humidity of more than 90%. The analysis of propanol may have the potential of being used as a shelf-life predictor. Cooking resulted in highly significant ($p < 0.01$) volatile loss.

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