Changes in the Volatile Profile of Oats Induced by Processing

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Samples of an Australian oat cultivar, Echidna, were pilot-scale processed. At each stage of the processing (raw oats, groats, kiln dried dehulled oats (KDHO), and rolled (flaked)) samples were removed for later sensory and GC−MS analysis of the flavor components. Mean taste panel scores from a trained taste panel were calculated according to attributes (cereal, burnt, toasted, floury, and yeasty). Attributes were generally similar for both KDHO and flaked oats except in the yeasty attributes. Panelists were able to differentiate between groats, KDHO, and flaked oats (raw oats were not included). The largest effects of heat processing were found for the attributes toasted and yeasty aroma; toasted, cereal, and yeasty flavor; and toasted and yeasty aftertaste. A multiorganoleptic sensor analyzer was able to differentiate all samples when the output was subjected to discriminant function analysis. A reintroduced sample was recognized with a confidence level better than 96%. Solid-phase microextraction (SPME) of headspace followed by GC−MS was used to identify volatiles after either dry or slurry heating. Several SPME fiber types were evaluated as to their ability to sorb oat volatiles. A 100-μm poly(dimethylsiloxane) SPME fiber was found to provide the best adsorption profile as measured by number of compounds sorbed and peak area response. A range of alcohols, aldehydes, alkyl benzenes, dienes, and ketones was identified in the processed samples.

Keywords: *Oats; processing; volatile profile; mass spectrometry; SPME*

INTRODUCTION

GC−MS studies have identified more than 100 compounds in the total volatiles fraction of oats, 35 compounds in headspace volatiles of oat groats (*1*), 45 volatiles obtained from vacuum steam distillation of rancid oat groats (*2*), and nearly 100 compounds in toasted oat groats (*3*). However, raw oats lack flavor, and the aroma/flavor that is associated with oat products requires the intervention of a heat process to develop (*1*−*4*). The effect of heat can be twofold: production of compounds responsible for the nut-like oaty aroma of cooked oats and/or inactivation of lipolytic enzymes.

Whole unblemished oats under normal storage conditions are stable, but the processes of grinding and/or flaking damage the grain and activate an endogenous lipolytic enzyme system that can cause rancid properties and poor flavor (*5*, *6*). Ideally, heating provides optimum stability of the product if the treatment is sufficient to inactivate lipolytic enzymes but mild enough to protect the natural antioxidants (*7*). Sjovall et al. (*4*) suggested that heat treatment generates reactions of metal ions with lipids and increases oxidation regardless of inactivation of the oxidative enzymes. The process also induces complex formation between lipids and gelatinized starch, and some breakdown of the lipids by heat can occur.

The effects of heat treatment and processing on the development of the oaty-nutty flavor of cooked oatmeal have also been investigated (*3*, *8*). Volatiles appeared (*3*) after the rolling and flaking stages of oat production and the associated heating, but from the concentration standpoint these volatiles were not much more abundant than the compounds discovered naturally in oat groats. Oxygenated components were the most significant neutral components in cooked oatmeal in which the flavor volatiles were mostly derived from lipid oxidation processes and interactions of reducing sugars and amino acids. These compounds included pentanal, furfural, heptanal, benzaldehyde, 1-octen-3-ol, 2,4-heptadien-1-al, phenylacetaldehyde, 3,5-octadien-2-one, 2,4-nonadien-1-al, γ-octalactone, 2,4-decadien-1-al, and γ-nonalactone. There was no single component to which "oaty" flavor might be attributed, but rather the flavor of cooked oatmeal was a blend of the impressions contributed by basic nitrogen heterocyclics and the neutral components.

This paper examines the effect of oat processing on the aroma profile of oat products. Isolation of the aroma compounds is an essential prerequisite to analysis but may contribute artifacts to the volatile profile (*3*). For instance, higher concentrations of N-heterocycles in basic fractions from volatiles isolated by Nickerson−Likens distillations of oatmeal were considered artifacts due to the severe conditions of isolation. Heydanek and McGorrin (*1*) found dramatic differences between the profiles and relative concentrations of volatiles obtained by vacuum steam distillation and dry vacuum isolation from groats. For example, a major peak such as limonene in the dry vacuum isolate represented a 10 ng g^{-1} concentration whereas hexanal in the hydrated isolation was present at 3−5 μg g^{-1}. The isolation procedure adopted in this study involved solid-phase microextraction (SPME) permitting the use of more gentle conditions for the recovery of volatiles.

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10.1021/jf0010127 CCC: $20.00
Published on Web 04/27/2001

MATERIALS AND METHODS

Oat Samples. Oats (10 kg, cv. Echidna) were obtained from Rennie, New South Wales, and they were processed at Uncle Tobys Research & Development Centre (Wahgunyah, Victoria, Australia). A sample was collected at each stage of the processing. Raw oats were dehulled with a pilot-scale oat dehulling machine. For kilning, live steam was injected into the column at approximately 102 °C to achieve an internal groat temperature of approximately 90 °C. The groats were held at that temperature for 45 min. Grain moisture content and temperature were then reduced in a series of steps so that the temperature was near ambient and the moisture content was around 9.5% when the kiln-dried dehulled oats (KDHO) exited the kiln. Rolled (flaked) oats were produced in a rolling process. The moisture content of the KDHO was raised by live steam from 9.5% to a flake moisture content of 11.5% and a temperature of 90 °C for 30 min. All samples were stored at 4 °C until analyzed.

Sensory Analysis. Trained panelists at Uncle Tobys Research & Development Centre evaluated samples of groats, KDHO, and flaked oats. Each ground sample (5 g) was consumed with milk (20 g). Mean sensory scores were calculated for each sample according to attributes (cereal, burnt, toasted, floury, yeasty) determined by 8 panelists (minimum 2 years experience) in a round-table discussion. Samples were not ranked for acceptability (hedonic scale) but for definable attributes. Attributes were scored on a line scale of 0−100 mm. Panelists were trained in twelve (1 h) sessions covering basic tastes and taste thresholds, fruit flavors, aromas, savory flavors, herbs and spices, essences, and texture. These sessions were followed by a further twelve (1 h) sessions of quantitative descriptive analysis (QDA) practice with on-going panel involvement evaluation.

Electronic Nose. Samples were analyzed in triplicate on an Alpha MOS Fox 4000 multi-organoleptic sensor analyzer, equipped with 18 metal oxide semiconductor sensors (Analytical Equipment Company, Ashburton, New Zealand). The sensors showed specific sensitivity to polar compounds, halogenated compounds, and solvents such as hexanol. Vials, each containing 2 g of sample, were loaded into an HS 50 headspace sampler and heated to 80 °C for 15 min. Following this, a headspace sample (2.5 mL) was removed and injected into the measuring chambers.

Analysis of Oat Volatiles. All water used for sample preparations was purified by a Modulab analytical model laboratory-research-grade water system. This system was designed to produce high quality water from pretreated reverse-osmosis feed through ultrafiltration membranes.

Chemicals used as standards were nonan-1-ol, undecan-1-ol, pentanal, trans-hex-2-enal, undecan-2-one, 1,2-diethylbenzene, 1,2,4,5-tetramethylbenzene, and 1-methylnaphthalene from Merck; 1-heptan-1-ol, hexanal, heptanal, octanal, nonanal, 2-furaldehyde, cyclopentanone, nonan-2-one, limonene, heptanoic acid, caproic acid, caprylic acid, palmitic acid, *n*-octane, *n*-nonane, *n*-decane, *n*-undecane, and *n*-dodecane from Sigma/Aldrich; cyclohexanol from BDH; and octan-1-ol and *n*-heptane from Ajax.

Three commercially available solid-phase microextraction fibers available from Supelco were examined for this study because of their reported (*9*, *10*) suitability to the analysis of volatile aroma compounds. These were poly(dimethylsiloxane) (PDS; 100 μm), carbowax/divinylbenzene (CDB, 70 μm), and divinylbenzene/carboxen/poly(dimethylsiloxane) (DCP, 50/30 μm). Septa for use in vials (20 mm), purchased from Supelco, were found to contain few volatile impurities.

Fiber Selection. An accurately known mass (approximately 100 mg) of dodecane (bp 216 °C), octadecane (bp 316 °C), hexan-1-ol (bp 158 °C), nonan-1-ol (bp 214 °C), undecan-1-ol (bp 243 °C), hexanal (bp 128 °C), octanal (bp 171 °C), nonanal (bp 192 °C), nonan-2-one (bp 195 °C), and undecan-2-one (bp 232 °C) was placed in a headspace vial (10 mL), sealed, and stored at ambient temperature prior to sampling. Each fiber was inserted through the vial septum into the headspace where adsorption occurred for 30 s. The volatiles were immediately thermally desorbed into the injection port of a Varian 3400 CX gas chromatograph equipped with a Saturn 2000 ion trap mass spectrometer and a DB-5 column (30 m × 0.25 mm i.d., 0.25 μm film thickness). The column temperature was initially held at 50 °C for 2 min, ramped from 50 °C to 110 °C at 10 °C min^{-1}, then 110 to 200 °C at 5.7 °C min^{-1}, and from 200 to 250 °C at 40 °C min^{-1}, with a 5-min hold at 250 °C. UHP helium with a linear velocity of 40 cm sec^{-1} was used as the carrier gas. The injector temperature was set at 250 °C with splitless injection mode.

The electron impact ionization (EI) mode with automatic gain control (AGC) was used for MS. The electron multiplier voltage for MS was 1850 V, AGC target was 25 000 counts, and filament emission current was 15 μA with the axial modulation amplitude at 4.0 V. The ion trap temperature was maintained at 250 °C and the manifold temperature was maintained at 60 °C. The temperature of the transfer line interfacing the GC and MS was set at 250 °C. Mass spectral scan time from *m*/*z* 35 to 450 was 0.8 s (using 2 microscans). Background mass was set at 45 *m*/*z*.

Dry Sampling. Samples (2 g) of raw oats, groats, KDHO, or flakes were placed in scintillation vials sealed with PTFE septum inserts. Vials containing samples were placed in an oil bath at 60 °C (*6*) with the SPME fiber inserted into the headspace above the oat sample. Adsorption was timed for 1 h. Cold extraction involved placing the sample (2 g) in a scintillation vial at ambient temperature and allowing adsorption to occur over 1 h. The effect of exposure time was

Table 1. Mean Sensory Scores for Oat Samples[a]

attribute	groats	KDHO	oat flakes	significance	*p*-value
aroma					
overall	50.8	53.2	44.5	**	0.008
cereal	32.0	40.7	33.0	***	0.000
burnt	43.7	0.0	0.0	***	0.000
toasted	0.0	35.5	24.7	***	0.000
yeasty	0.0	0.0	20.2	***	0.000
flavor					
overall	52.7	54.8	46.2	*	0.073
burnt	43.5	0.0	0.0	***	0.000
toasted	0.0	39.5	22.0	***	0.000
floury	34.8	15.3	18.0	**	0.005
cereal	0.0	36.5	28.2	***	0.000
yeasty	0.0	0.0	25.3	***	0.000
aftertaste					
burnt	34.0	1.2	2.2	***	0.000
toasted	0.0	31.8	16.0	***	0.000
yeasty	0.0	0.0	21.3	***	0.000

[a] Significance levels: *p*-value 0.000−0.001, *** = 0.1% significance level; 0.001−0.010, ** = 1% significance level; 0.010−0.050, * = 5% significance level.

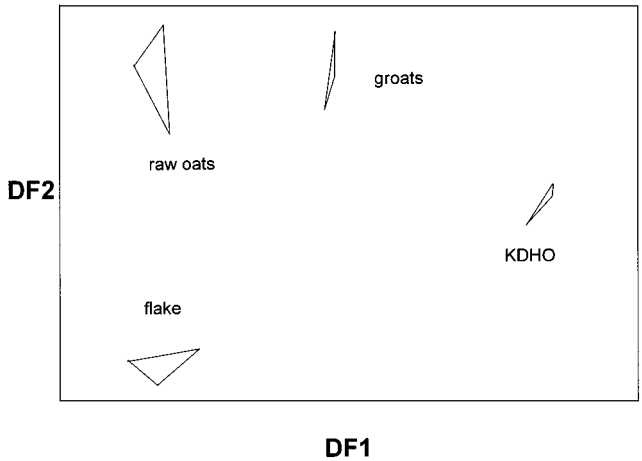

Figure 1. Discriminant function analysis of data obtained by electronic nose using DF1 (92.33%) and DF2 (6.32%) to distinguish between raw oats, groats, KDHO, and flaked oats.

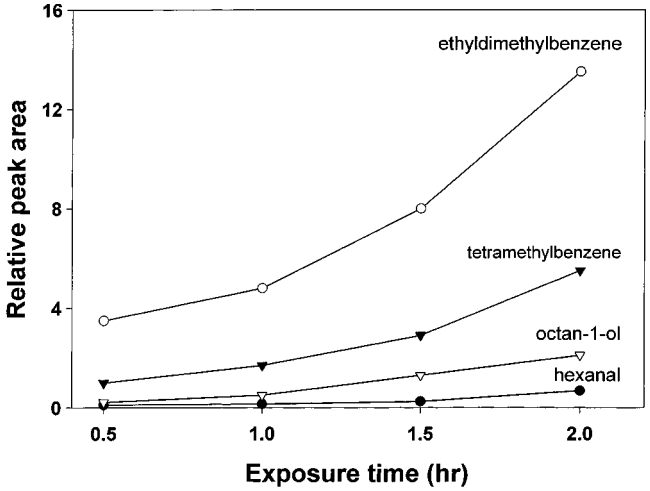

Figure 2. Effect of exposure time on the amount of solute adsorbed from flaked oats by a DCP fiber. Data are shown for four selected compounds prior to equilibration time.

determined by varying the time the fiber was exposed to the headspace of flaked oats.

Slurry Sampling. Slurry sampling was performed in the same manner except that oat samples (2 g) were slurried with distilled water (2 mL) prior to being heated at 60 °C and exposed to the SPME fiber. The use of boiling slurries reduced the concentration of volatiles being adsorbed onto the fiber due to steam condensation. Stirring of the slurry was achieved with a laboratory stirrer and glass stirring rod with paddles rather than the conventional magnetic stirrer because of the high viscosity of the oat slurries.

GC−MS. Oat volatiles isolated by either dry or slurry sampling were analyzed by thermal desorption in the injection port of a Varian 3400 CX gas chromatograph coupled with a Saturn 2000 ion trap mass spectrometer using the same conditions as described for fiber selection but with a BPX-5 column (30 m × 0.25 mm i.d., 0.25 μm film thickness, SGE, Australia).

RESULTS AND DISCUSSION

Raw oats, groats, KDHO, and flaked (rolled) oats were analyzed by taste panel, electronic nose, and GC−MS following SPME from dry-heated and slurried samples in order to assess aroma development during the course of processing and the ability of the techniques to differentiate between samples at the different processing stages.

Sensory Analysis. Quantitative data are presented in Table 1 for each of the samples (raw oats were not included) for each attribute. All attribute values with the exception of two were significant at the 0.1% level. The attributes were generally similar for both KDHO and flaked oats with the exception of the yeasty attributes where the flaked oats had the only significant impression. This suggests that the further steaming and heating involved in production of the flaked oats creates a greater yeasty flavor. Nevertheless, panelists were able to differentiate between the groats, KDHO, and flaked oats. The aroma attributes were generally directly related to their corresponding flavor attributes. For example, the flaked oats gave a yeasty flavor of 25.3 and a yeasty aroma of 20.2, but the other samples were zero in both cases. The largest effects of heat processing were found for the attributes toasted and yeasty aroma; toasted, cereal, and yeasty flavor; and toasted and yeasty aftertaste. KDHO was perceived as having the strongest overall aroma and flavor. The burnt aroma and flavor of the groat sample is attributed to the high oil content of the groats that caused it to stick to the grinder.

Electronic Nose Analysis. The raw oats, groats, KDHO and flakes were also analyzed by electronic nose, and the data were analyzed using principal component analysis (PCA) (not shown) and discriminant function analysis (DFA) (Figure 1). PCA provided good separation of raw oats, groats, and KDHO with 91.4% of the

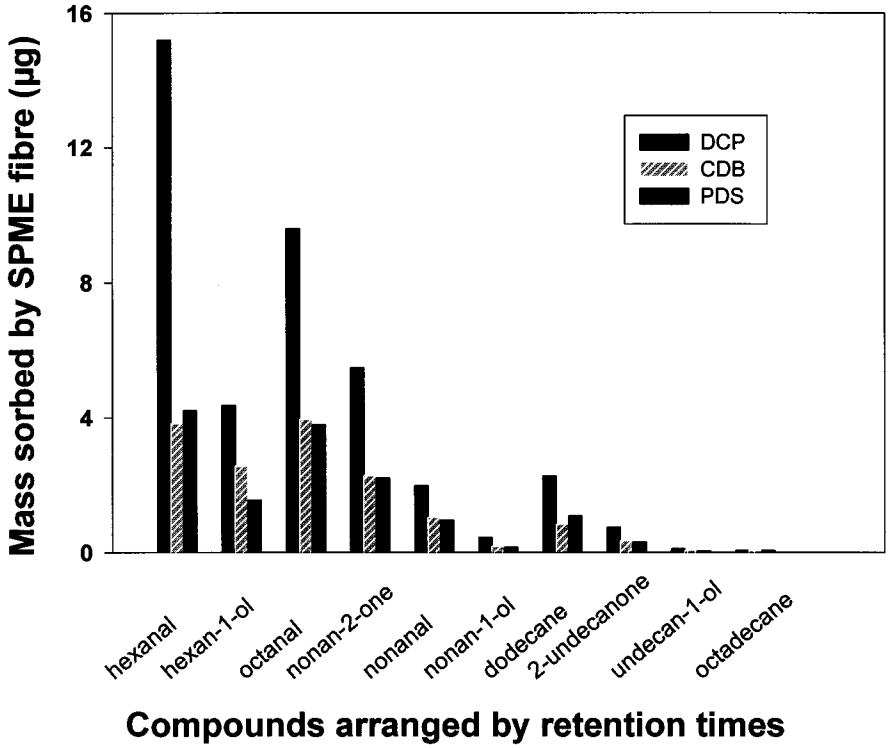

Figure 3. Comparison of the amount of compound adsorbed by the PDS, CDB, and DCP fibers. Data have been corrected for detector response.

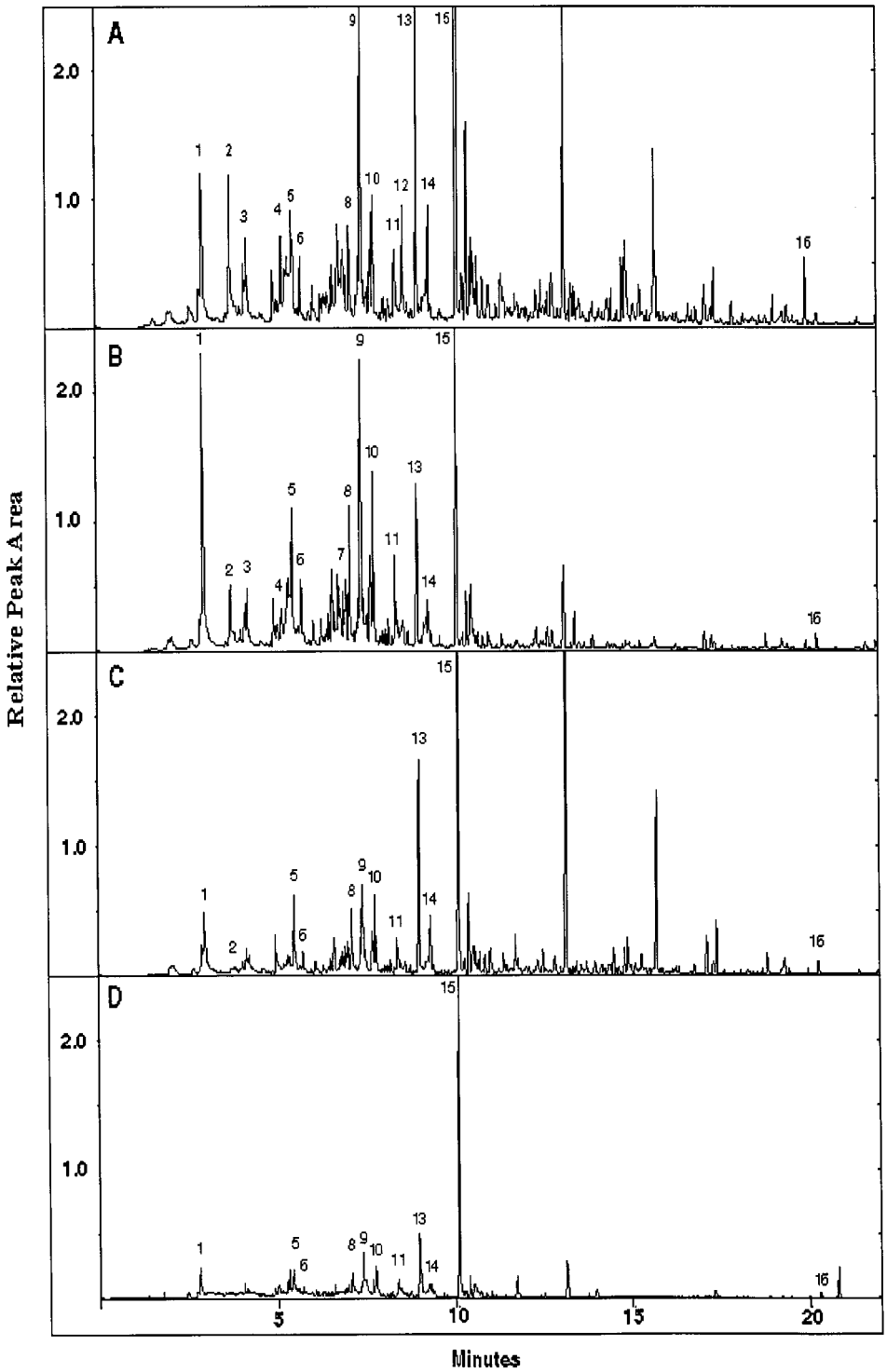

Figure 4. Chromatograms comparing the volatile profiles of (A) raw oats, (B) groats, (C) KDHO, and (D) flaked oats. Data were obtained by warm extraction from dry samples. Mass spectral detection was achieved by EI ionization. For peak identities, refer to Table 2.

variation accounted for by PC1 and a further 5.3% accounted for by PC2. However, flaked oats and raw oat classes overlapped, and the discrimination between these classes was enhanced by using DFA. Because of the small number of samples, DFA was possible with the use of only five sensors. This is more desirable as it reduces the number of independent statistical variables. Cross validation was executed by the removal of one sample label, the sample was then recognized with a confidence level better than 96%.

Additional samples were introduced to the electronic nose as a further sample of KDHO (cv. Echidna) and a consumer complaint sample previously identified by the trained panel as having a perfume or soapy taste. The system successfully grouped the KDHO sample with that previously analyzed and separated the consumer complaint sample from all others but with a close link to the raw oats. This is presumably a consequence of the high oil content of the raw oats and a tendency to rancidity.

Analysis of Aroma Compounds. *Fiber Selection.* Control of experimental variables is critical in head-

Table 2. Compounds Identified in Oat Samples by SPME Using Warm Extraction from Dry Samples[a]

peak no.	retention time (min)	Kovat's retention index	compound	raw oats	groats	KDHO	oat flakes
	2.58	<700	pentan-1-ol	*	*		
1	2.92	808	hexanal	C	C	C	C
2	3.72	875	hexan-1-ol	C	C	C	
3	4.18	916	heptanal	C	C		
4	5.18	978	heptan-1-ol	C	C		
	5.30	986	benzaldehyde	C		C	C
5	5.49	999	2-pentylfuran	C	C	C	C
6	5.70	1012	octanal	C	C	C	C
	6.13	1039	limonene	C		C	
	6.62	1069	dimethylethylbenzene	L	L	L	L
7	6.80	1080	octan-1-ol		C		
8	7.11	1099	dimethylethylbenzene	L	L	L	L
9	7.40	1127	nonanal	C	C	C	C
10	7.78	1135	tetramethylbenzene	L	L	L	L
	7.86	1139	tetramethylbenzene	C	C	C	C
11	8.39	1167	tetramethylbenzene	L	L	L	L
12	8.63	1180	nonan-1-ol	C			
13	9.00	900	dodecane	C	C	C	C
14	9.32	1216	decanal	C	C	C	C
	9.71	1235	E,E−2,4-nonadienal		C		
15	10.15	1257	dimethylethylbenzene	L	L	L	L
	11.04	1302	2-undecanone	C	C	C	C
	11.85	1338	E,E−2,4-decadienal		C		
	13.39	1410	6,10-dimethyl-2-undecanone	S	S	S	
	15.80	1523	tridecanal	S	S	S	
16	20.01	1728	pentadecanal	L	L	L	L

[a] "*" indicates observed in cold extractions only. C = Identification based on correspondence of retention time and mass spectrum with genuine standard. L = compound previously observed in oats and identified using a mass spectral library with a quality match exceeding 84%. S = compound identified using a mass spectral library with a quality match exceeding 84% and not previously reported in oats.

space SPME. Exposure time is important; and the amount of solute adsorbed increased as exposure time was increased (Figure 2), although equilibration time varied greatly. Thus, shorter sampling times show reduced sensitivity as compared to exhaustive SPME (at the relevant equilibration times). Nevertheless, shorter sampling in SPME better approaches conventional static headspace analysis (*10*) and minimizes extraction of matrix components and hence the opportunity for artifactual components. At the same time, the fibers were expected to show high affinity for pyrazines (*10*) that are reported as important components of cooked flavors (*3*). Hence, a 1 h isolation time was selected as a compromise between analysis time, sensitivity, and minimizing artifactual components.

Fiber type is also important, and Figure 3 compares the masses of different solutes adsorbed by the three fibers. Solute volatility was clearly important in determining adsorption as seen by comparing compounds with the same functional group but differing in carbon chain length. For compounds of similar volatility (as determined by boiling point and by interpolation) the extent of adsorption decreased as ketone > aldehyde > alcohol. These trends were common to the three fibers. Considering individual solutes, greatest adsorption occurred on the DCP fiber followed by the CDB fiber, with lowest adsorption on the PDS fiber. This is probably in part a reflection of the amount of adsorbent contained on the fiber. The nominal masses of adsorbent calculated from the fiber dimensions (PDS, 1 cm length, 1.0×10^{-4} m radius; CDB, 1 cm length, 1.2×10^{-4} m radius; DCP, 2 cm length, 1.6×10^{-4} m radius), coating thickness (100, 65, 50, and 30 μm, respectively), and density of the adsorbent (assumed constant for all phases at 0.895 g mL^{-1}) in a fashion analagous to that used in GC capillary columns, were 2.1×10^{-10} g, 3.0×10^{-10} g, and 1.2×10^{-9} g for the PDS, CDB, and DCP fiber, respectively. On the assumption of these calculated masses, there would appear to be minimal selective adsorption but rather that the adsorption per unit mass of adsorbent was greatest on the nonselective PDS fiber. Nevertheless, the DCP fiber showed greatest absolute adsorption of all solutes because of its greater fiber length and coating thickness, and it was used in analyses of oat samples.

Oat Aroma Profile. Headspace SPME provided a rapid method for analysis of oat volatiles using small samples (2 g) of both dry oats and slurries. This is an advantage in breeding trials where the amount of sample is limited. In previous studies on oats, Heydanek and McGorrin (*1*) used large sample sizes of up to 8 kg to obtain sufficient volatiles from oat groats via vacuum distillation. The SPME technique was applicable to a wide range of volatiles with Kovat's retention indices ranging from 700. Blank runs produced several peaks attributed to the fiber but at levels several orders of magnitude below that arising from sample components.

Figure 4 compares the volatile profile obtained by warm extraction of the various stages of processing. The amount of volatiles recovered by warm extraction from dry oats was 1−2 orders of magnitude greater than that achieved with cold extraction. However, the same range of compounds was observed in both cases suggesting that the compounds were not artifactual and formed by heating during the isolation process. Pentan-1-ol was isolated by cold extraction of raw oats and groats but not by warm extraction. This volatile compound may have been lost in the warm extraction. Similar profiles were observed with slurry sampling. The concentration of volatiles decreased during the various stages of processing from raw oats to oat flakes. This may be considered unusual as the most distinct flavors came from the final products, which have a strong nutty/oaty

flavor. However, Sjovall et al. (*4*) found a 90% loss of volatiles during extrusion processing.

Compounds identified in the oats samples are listed in Table 2. The compounds identified were mainly aldehydes and alcohols with hexanal, 2-pentylfuran, several isomers of dimethylethylbenzene and tetramethylbenzenes, nonanal, and decanal the most prominent peaks in the flake sample. The presence of aldehydes has been attributed (*3*) to reactions occurring in boiling water heat treatment. However, this would not seem to be the case in the present study as these compounds were present in raw samples before heat treatment and they were also detected by cold extraction. It seems more likely that the aldehydes, including benzaldehyde arose (*3*) from interactions of reducing sugars and amino acids. Alternatively, the presence of C_5 and C_6 alcohols and aldehydes as also reported in oat groats by Heydanek and McGorrin (*1*) was of particular significance, as they typically relate to lipoxygenase and oxidoreductase activity. Heydanek and McGorrin (*2*) suggested that hexanal would be a good indicator of the development of oxidative rancidity from oat oil fatty acids. However, hexanal is present in flake samples that had an acceptable flavor character for human consumption. Therefore, rancidity in oat containing foods would be a function of the change of concentration of hexanal rather than its absolute presence (*2*). Consistent with this interpretation, hexanal was present at twice the level in oat husks than in raw oats and 30 times greater in oat husks than in flaked samples. This suggests that the concentration of hexanal observed could be associated with oxidative rancidity in the raw oats and more so the husk. This is also seen in the analysis by electronic nose where a rancid sample was classified with the raw oats. The heat process could then be considered as deactivating the oxidation process.

A similar trend is observed for hexan-1-ol which was abundant in the husk and raw oats but was halved in the groats. The level was further reduced in KDHO and it was not detected in the flakes. In their study, Molteberg et al. (*6*) found that only hexanal and hexan-1-ol showed major changes due to heat treatment. Increases in hexanal, however, do not explain the flavor changes associated with heating, as hexanal is usually related to a green flavor, not the flavor observed. The same authors (*6*) noticed a reduction in the level of hexan-1-ol due to heating and suggested that there was an improvement in flavor associated with the heating and loss of this compound. This could have happened in the present study as flavor was improved in the stages where the compound was reduced. The compound 2-pentylfuran was present in every sample, and as with most of the compounds there was a reduction in abundance in the KDHO and more so in the flake sample. This compound is also related to rancidity when present in high concentrations.

Sensitivity in SPME is very dependent on the compound analyzed (*10*), but nonpolar compounds are easily detected at the ng g^{-1} level accounting for the levels of dodecane and substituted benzenes in the volatile profile. In contrast, substituted pyrazines that are often associated with "cooked" flavor (*3*) were not detected in the present study, contrary to expectations based on their high affinities for SPME fibers (*10*). These compounds have retention indices (*11*) within the range examined in this study and typical detection limits varying between 0.5 and 1 μg g^{-1} as pure components in solution. This suggests that pyrazine levels in the oats (2 g sample) were below 250−500 ng g^{-1}, although competition for adsorption on fibers is well documented (*10*) and this may complicate the calculation of levels in the oats. Sjovall et al. (*4*) were also unable to find such compounds. Consequently, the origin of the "cereal" flavors is unclear although the volatile profile can also distinguish between the various stages of processing.

ACKNOWLEDGMENT

The authors gratefully acknowledge the assistance of Uncle Tobys Ltd. The assistance of David Fotheringham, Analytical Equipment Company (Ashburton, Victoria), in providing electronic nose data is gratefully acknowledged.

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Received for review August 14, 2000. Revised manuscript received February 12, 2001. Accepted February 13, 2001. This study was conducted with the support of a grant from the Grains Research and Development Corporation of Australia.

JF0010127